

# DEVELOPMENT OF ADVANCED MATERIALS COMPOSITES FOR USE AS INSULATIONS FOR LH<sub>2</sub> TANKS

**FINAL REPORT**  
**MAY 1972 TO APRIL 1973**

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C.R. LEMONS  
O.K. SALMANS

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Prepared under Contract NAS 8-22973  
by Materials and Methods Research  
and Engineering Department  
McDonnell Douglas Aerospace Company  
Huntington Beach, California  
for the  
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**PREFACE**

This document reports the results of Phase III of the NASA Marshall Space Flight Center program entitled, "Development of Advanced Materials Composites for Use as Insulation for LH<sub>2</sub> Tanks." This work is being conducted under NASA Contract NAS8-25973. Mr. W. E. Hill serves as principal contracting officer representative. This final report covers the period from 1 May 1972 to 1 February 1973.

The Program Manager is O. K. Salmassy and the Principal Investigator is C. R. Lemons. This program is under the overall technical direction of R. F. Zemer, Deputy Director, Materials and Methods - Research and Engineering.



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## ABSTRACT

A study of thread-reinforced polyurethane foam and glass fabric liner, serving as internally bonded insulation for NASA Space Shuttle LH<sub>2</sub> Tanks, is reported. Emphasis was placed on an insulation system capable of reentry and multiple reuse in the Shuttle environment. The optimized manufacturing parameters associated with each element of the composite are established and the results, showing successful completion of subscale system evaluation tests using the shuttle flight environmental requirements, are given.



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## Section 1

### INTRODUCTION

The primary objective of this program has been to develop reliable advanced material composites for a minimum-weight internal insulation that will provide adequate protection of NASA Space Shuttle LH<sub>2</sub> tanks for up to 100 and preferably 200 missions. Emphasis has been placed upon developing a concept that will not be adversely affected by LH<sub>2</sub> tank outer-surface temperatures of up to 177°C (350°F).

The basic approach of this program has been to develop a modification of the MDAC/NASA-developed S-IVB-3D foam insulation for application to the more severe environment of the Space Shuttle. Phase I of this program was successful in developing the basic material. An improved composite was developed and its performance verified.

The baseline composite selected from Phase I of this program consisted of three-dimensionally (3D) reinforced, heat-stabilized BX-249N foam, with a liner of 878/CL resin-impregnated 116-glass cloth and a tank-wall adhesive of L211A/LZ resin.

Objectives of Phase II included optimization of the baseline composite and analysis of Space Shuttle vehicle environments not considered previously. Tank insulation fabrication and installation procedures directly applicable to the Space Shuttle were established. During Phase II, an as-machined metal surface with a chromic-acid anodized coating was selected as the baseline tank-wall condition (Task 7). Also during Phase II, as-molded BX-251A-3D form with silane (EC 3901) primed fibers was substituted for BX-249N-3D-S as the baseline reinforced foam system.

Because of functional similarities between S-IVB and Shuttle internal insulation requirements, the performance requirements established for the S-IVB system were used during Phase I as the basis for material developments(Reference 1). Phase B Shuttle studies were subsequently completed along with extension studies (Reference 2).

During the Shuttle Phase B studies, emphasis was shifted from a completely reusable system to analysis of an Orbiter with external expendable tanks (Reference 3). Under a recent expendable tank concept, the Orbiter carries its hydrogen propellant in one external tank. The tank would have a smooth wall with reinforcement in some areas. None of the insulation requirements set forth in Reference 1 was altered for this study by these more recent system considerations.

The objectives of Phase III were to optimize the weight of the Phase II system further and to verify the cryogenic thermostructural performance of the Phase II composite and the lightweight composite resulting from Phase III.

## Section 2

### EXPERIMENTAL PROGRAM

The Phase III experimental program was divided into the following tasks:

- Task 1 - Literature Survey**
- Task 2 - Lightweight Foam Optimization**
- Task 3 - Lightweight Gap Filler For Joints**
- Task 4 - Minimum Tank-wall Adhesive Weight**
- Task 5 - System Evaluation Tests**

The approach to Phase III was to proceed from the development of a composite based on large-scale fabrication and installation procedures with the cryogenic verification of the resulting performance. The approach of Phase III went one step further and sought the development and cryogenic verification of a lighter-weight and a lower-cost version of the Phase II composite.

#### **2.1 LITERATURE SURVEY (TASK 1)**

The objective of this task was to maintain a comprehensive survey of the technical literature dealing with insulation systems, test methods, Shuttle technology, materials and processes, and other areas related to the requirements of this program. The survey should help ensure that the optimum selection of materials and fabrication procedures is made. Lists of pertinent information sources have been reported in Monthly and Quarterly Reports.

#### **2.2 LIGHTWEIGHT FOAM OPTIMIZATION (TASK 2)**

At the completion of Phase I of this development program, the baseline 3D foam selection consisted of a polyurethane foam manufactured by Nopco Chemical Company as BX-249N reinforced with the same glass yarn three-dimensional array as used for Saturn S-IVB 3-D foam. This BX-249N-3D foam has a density of 56 kg/m<sup>3</sup> (3.5pcf) and compares favorably with the

S-IVB 3D foam having a density of  $83.2 \text{ kg/m}^3$  (5.2 pcf) relative to thermal and structural efficiency when used as internal cryogenic insulation. By exposing the slices of BX-249N-3D foam to an oven-bake cycle of  $177^\circ\text{C}$  ( $350^\circ\text{F}$ ) for 16 hours prior to bonding inside the  $\text{LH}_2$  tank, this foam would remain relatively stable during subsequent exposures to elevated temperatures. The heat-stabilized BX-249N-3D-S foam successfully completed the 1-meter (3-ft) dome test program for strain compatibility conducted at tank wall temperatures of  $-253^\circ\text{C}$  ( $-423^\circ\text{F}$ ) ( $\text{LH}_2$ ) and at  $177^\circ\text{C}$  ( $350^\circ\text{F}$ ) simulating the Shuttle  $\text{LH}_2$  tank environment at launch and through aerodynamic heating profiles.

During Phase II of this development program, an improved polyurethane foam, also manufactured by Nopco Chemical Company and identified as BX-251A, was developed as the matrix for the 3D glass yarn array. The advantages of BX-251A-3D were lighter weight,  $44 \text{ kg/m}^3$  (2.75 pcf) and capability of dimensional stability when exposed to temperatures from  $-253^\circ\text{C}$  ( $-423^\circ\text{F}$ ) to over  $177^\circ\text{C}$  ( $350^\circ\text{F}$ ) without any preconditioning process cycles.

With COR concurrence, BX-251A-3D was selected as the baseline 3D foam for additional 1-m (3-foot) dome testing required for Phase III of this program.

Detail testing to determine the effectiveness of manufacturing operations in the 1-m (3-ft) domes, Tasks 3 and 4, used the new BX-251A polyurethane foam in the glass thread three-dimensional array. These reinforced foam blocks were fabricated using the following procedures in subsection 2.2 and 2.2.2 as derived from Phase II of this development program.

#### 2.2.1 Glass Thread Array

- Wind ECG-150-1/0 3.8 glass yarn on aluminum frames, depicted in Figure 2-1, to provide a discrete spacing of  $0.475 \text{ cm}$  (0.187 in.) for all the X and Y direction threads.
- Cross-stack the X and Y frames horizontally to a height of  $20 \text{ cm}$  (8 in.)

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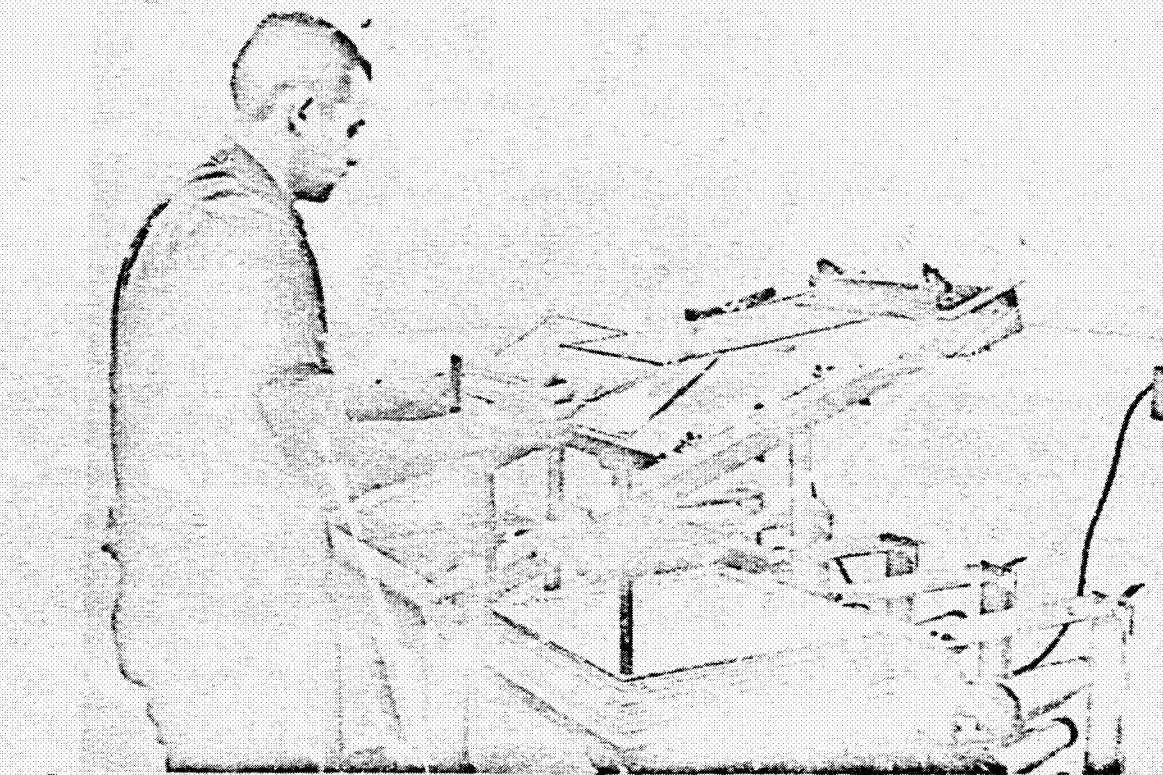


Figure 2-1. Winding X and Y Frames

- Weave ECG-150-3/2 3.8 glass yarn vertically into the X-Y array as shown in Figure 2-2 and hold in place 0.475cm (0.187 in.) on center by using steel rods at the top and bottom faces.
- Sprinkle EC-3901 (3M) primer over the top of the array and allow the primer to coat all threads by capillary action.
- This X, Y, and Z glass-thread array is now ready for foam injection.

#### 2.2.2 Foam Mixing Machine Operation and Injection of Foam into 3D Thread Array (Figure 2-3)

- Stabilize foam ingredients to  $28 \pm 2^\circ\text{C}$  ( $80 \pm 3^\circ\text{F}$ ) and recirculate for 1 hour prior to calibration pouring.
- Adjust hydraulic pump pressures to achieve a ratio of 41R to 59T within  $\pm 2.5$  percent "T".

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Figure 2-2. Weaving Machine for Z Fibers

- Adjust blending rotor speed between 5,000 and 6,000 rpm.
- Dispense 2,146 grams of liquid foam with a 10-sec pour time onto a polyethylene pan located just below the outlet nozzle so that air does not become entrained in the blended foam. Then lower the filled pan to rest against the bottom of the mold.
- Place the frame-supported glass-thread array over the mold and clamp in position.
- Allow the foam to rise to the top of the frames and set hard.
- Remove the metal frames from the 3D block 20 x 30 x 30 cm (8 x 12 x 12 in.) and slice the reinforced foam to the thickness required for tank insulation as shown in Figure 2-4.

In the course of Phase III, the properties of RX-251A-3D were evaluated and its processing optimized, as noted above, in an attempt to achieve a material composition and construction necessary for reliable use. A

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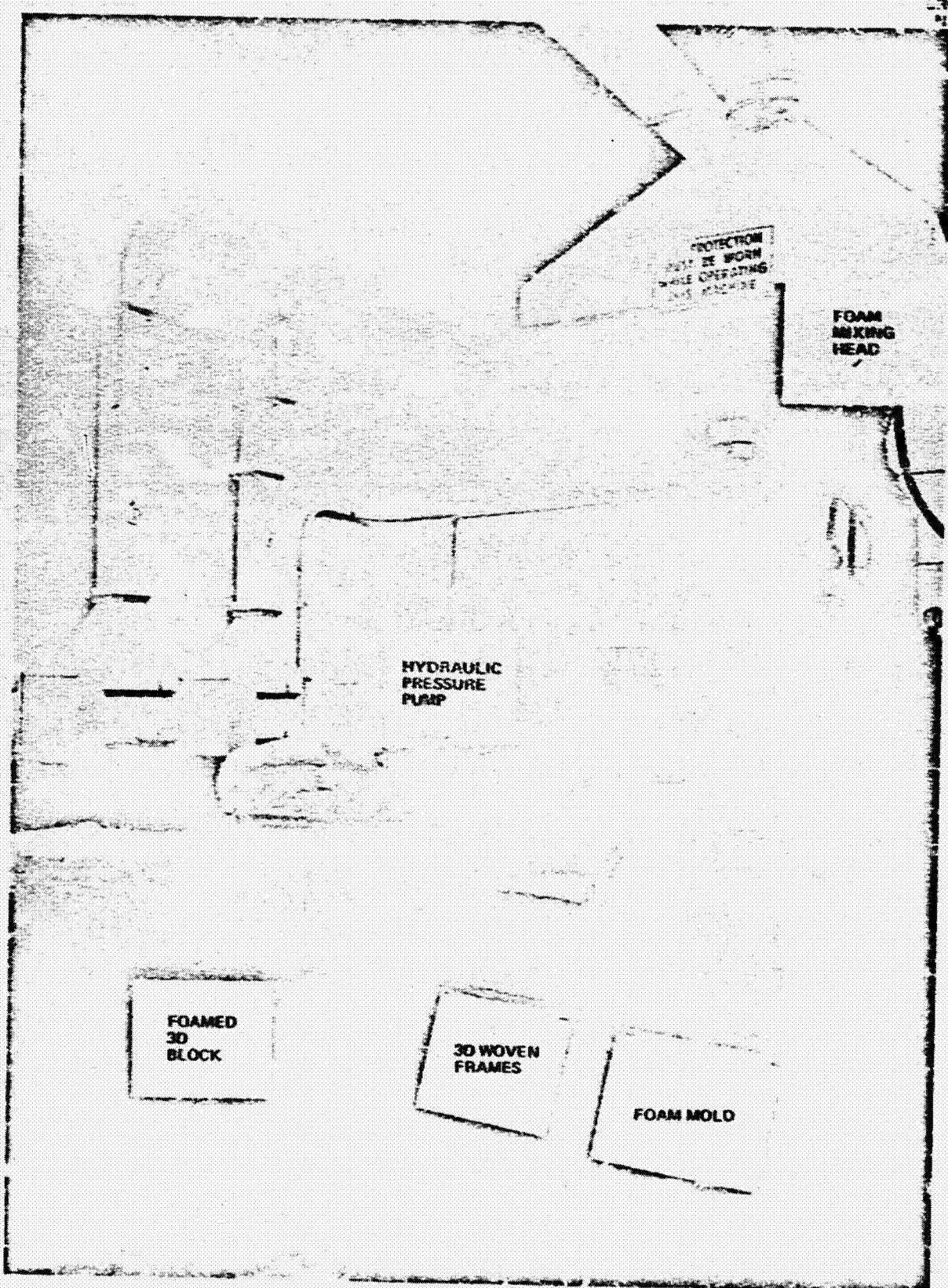


Figure 2.3. 3D Foam Fabrication Setup

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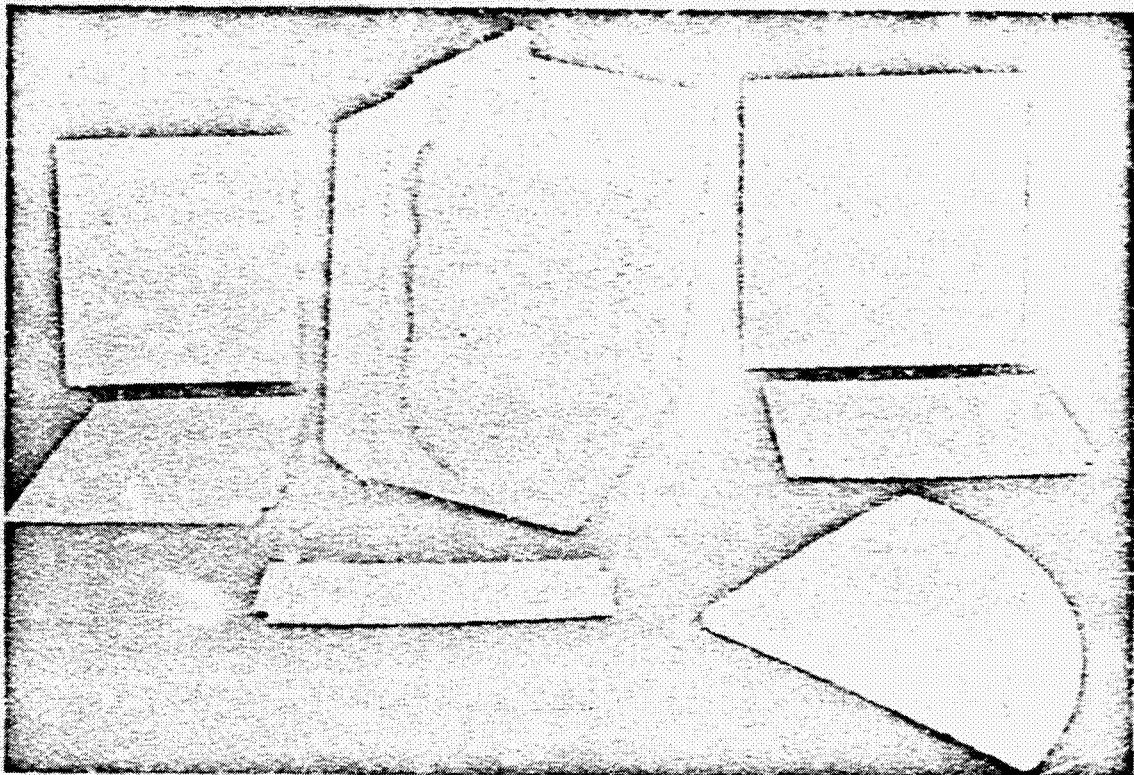


Figure 2-4. 3D Foam Panels

summary of some of the physical and mechanical properties of BX-251A-3D is given in Table 2-1. Derived criteria for foam acceptance are given in the detailed specifications submitted with this report.

During the Phase III effort, production of BX-251A polyurethane foam was interrupted by a change in management at Nopco Chemical Company, New Jersey. The parent company, Diamond Shamrock Corporation divested itself of Nopco Chemical Company which was acquired by Stepan Chemical Company, Chicago, Illinois.

### 2.3 LIGHTWEIGHT GAP FILLER FOR JOINTS (TASK 3)

The objective of this task was to develop and evaluate low density material for filling joints in 3D foam insulation.

**Table 2-1**  
**PROPERTIES OF BX-251A AND WITH 3D**

	Neat BX-251A Polyurethane Foam	3D thread reinforced BX-251A
Foam density	24 kg/m <sup>3</sup> (1.5 pcf)	44 kg/m <sup>3</sup> (2.75 pcf)
Tensile 25°C (77°F)		
Parallel to rise	0.21 MN/m <sup>2</sup> (30 psi) at 4-percent elongation	1.38 MN/m <sup>2</sup> (200 psi) at < 2-percent elongation
Perpendicular to rise	0.12 MN/m <sup>2</sup> (17 psi) at 6-percent elongation (foam rupture)	X-Y and Z direction (thread bond rupture)
Compression 25°C (77°F)		
Parallel to rise	0.14 MN/m <sup>2</sup> (20 psi) at 2-percent deforma- tion	0.45 MN/m <sup>2</sup> (65 psi) at < 3-percent defor- mation
Perpendicular to rise	0.048 MN/m <sup>2</sup> (7 psi) at 4-percent deforma- tion	Z threads rupture
0.05 MN/m <sup>2</sup> (8psi) at 10-percent deforma- tion		
Shear 25°C (77°F)		
Parallel to rise = Z direction Modulus (G)	0.09 MN/m <sup>2</sup> (12 psi) 0.80 MN/m <sup>2</sup> (116 psi)	0.46 MN/m <sup>2</sup> (66 psi) 3.80 MN/m <sup>2</sup> (550 psi)
Weight loss	7 percent	6 percent
16 hr at 177°C (350°F)		

Studies of insulation panel joints in Phase II indicated that the butt joints may be as acceptable as the baseline S-IVB shiplap joint for use in the Shuttle application. The use of butt joints would significantly reduce installation time. Using either the butt joint or the shiplap joint, it may be desirable to accept gaps between mating tile surfaces in order to reduce machining tolerances and thereby reduce machining and installation costs. If so, it may be desirable to fill the gaps with a low-density material to reduce heat losses and improve the mechanical integrity of the insulation system.

The thermal and structural analysis presented in the Phase II Summary Report (June 1972) indicates no adverse effects will result from open gaps up to 0.15-cm (0.06-in.) in width between 3D foam tile, and this joint may have shiplap edges, stepped edges, or straight-butt edges. The adhesive deposited on the face of the 3D foam tile using the adhesive mixing machine will normally string over the edge of each tile and provide a joint bond without the addition of a gap filler if the edges are within 0.15 cm (0.06 in.) of each other. Therefore, the gap filler is needed only in the event the gap width exceeds 0.15 cm (0.06 in.).

The probability of a large number of such gaps occurring during insulating a large tank could introduce a weight control problem unless the gap filler was considerably lighter in weight than solid adhesive such as Lefkoweld 211A/LZ. The gap filler must possess the following properties:

- A. Light Weight — The use of solid adhesive to fill gaps that might occur with insulating a large Shuttle tank could add as much as 46 kg (100 lb) to the vehicle insulation weight. Preliminary tests described in the Phase II Report (Ref. 4) indicate the feasibility of reducing this weight by 50 percent by using a syntactic foam mixture between tile edges.
- B. Strain Compatibility in LH<sub>2</sub> — The gap-filled joint should not create stress concentrations in the glass liner resulting in ruptures when exposed to LH<sub>2</sub>.

- C. Elevated Temperature Resistance - The gap filler should not expand, outgas, or lose structural integrity when exposed to 177°C (350°F) as part of the insulation composite.
- D. Chemical Compatibility - The "wet" gap filler will be cured in contact with "wet" 211A/LZ adhesive and Epon 828/CL glass liner, and must not adversely affect the properties or processing of either resin system.
- E. Low-Cost Filling Procedure - The gap filler should have the same, or longer, catalyzed working life as 211A/LZ or Epon 828/CL resins used to bond the insulation and liner. The gap filler should be easily mixed and dispensed as needed during the tile laying operations.

Specimens for tests to determine cryogenic strain compatibility were fabricated (Figure 2-5). Three types of joints were used to allow the gap filler 0.3-cm (0.12-in.) width to be stressed in the longitudinal direction and lateral directions while held in restrained position and immersed in cryogen.

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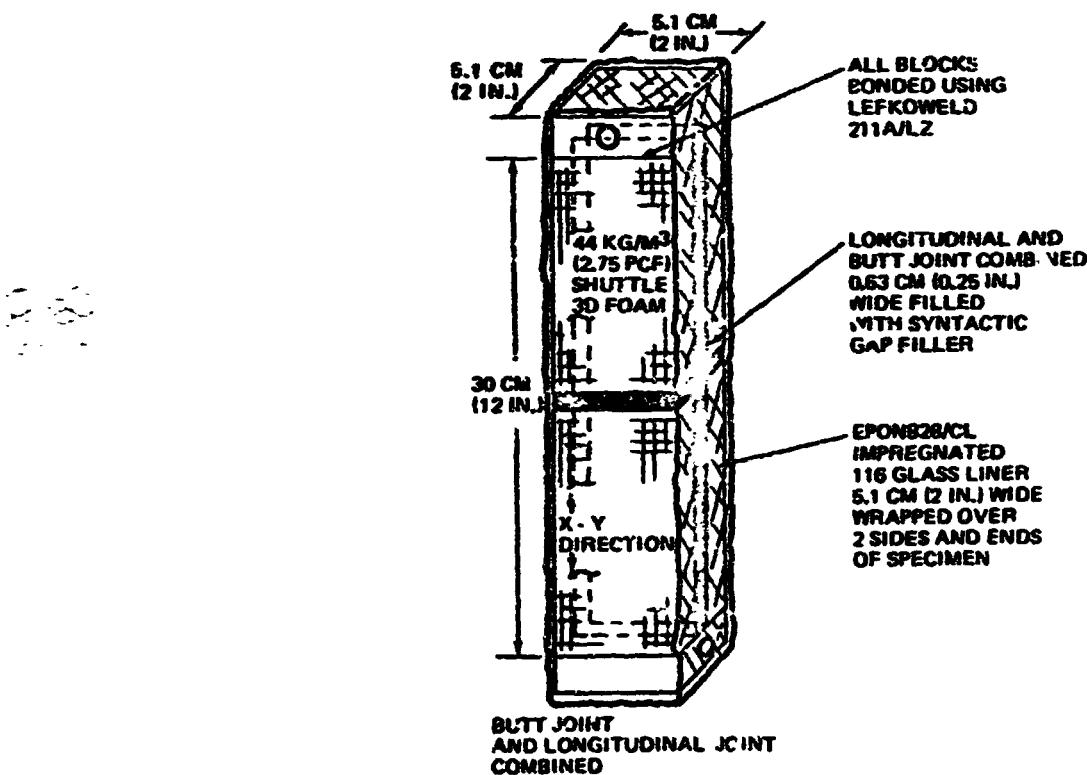


Figure 2-5. Joint Test Configuration 3D Foam

Epon 828/CL epoxy resin filled with BJO-0930 microballoons (phenolic) and having a density of  $0.53 \text{ g/cm}^3$  (33 pcf) was used in one set of specimens. Polyurethane resin U-135/MOCA/with 1-percent Z-6040 filled with BJO-0930 microballoons (phenolic) having a density of  $0.49 \text{ g/cm}^3$  (30.4 pcf) was used in another set of specimens. Lefkoweld 211A/LZ adhesive was used as reference in another set of specimens. Lefkoweld 211A/LZ bonded joints were used successfully in the 1-m (3-ft) dome cycling tests conducted in Phase I of this development program and were tested in this series to provide a basis for comparison.

The restrained contraction load in  $\text{LN}_2$  was determined on each gap filler material using the test setup described in Figure 2-6. Results of the first series of these tests are given in Table 2-2.

All the materials tested as joint fillers without a glass liner bonded over the joint developed contraction cracks when held restrained in the longitudinal direction and chilled to  $-196^\circ\text{C}$  ( $-320^\circ\text{F}$ ) ( $\text{LN}_2$ ). The joint filler, however, did not separate from the edges of the 3D foam during chilldown and the composite was capable of withstanding an applied load 50-percent higher than the load induced by thermal contraction.

The Lefkoweld 211A/LZ bonded butt joints developed a higher rupture strength than achieved using microballoon filled formulations, as was expected; however, Lefkoweld 211A/LZ developed contraction cracks just as readily as the other formulations. No significant structural advantage was realized by using the silanepolyurethane resin as compared to the epoxy resin formulation with microballoons. A distinct advantage is attached to the use of epoxy resin joint filler because the glass liner to be bonded over the joint will also be impregnated with the same epoxy resin and thus provide a completely compatible curing system in conjunction with the epoxy tank-wall adhesive.

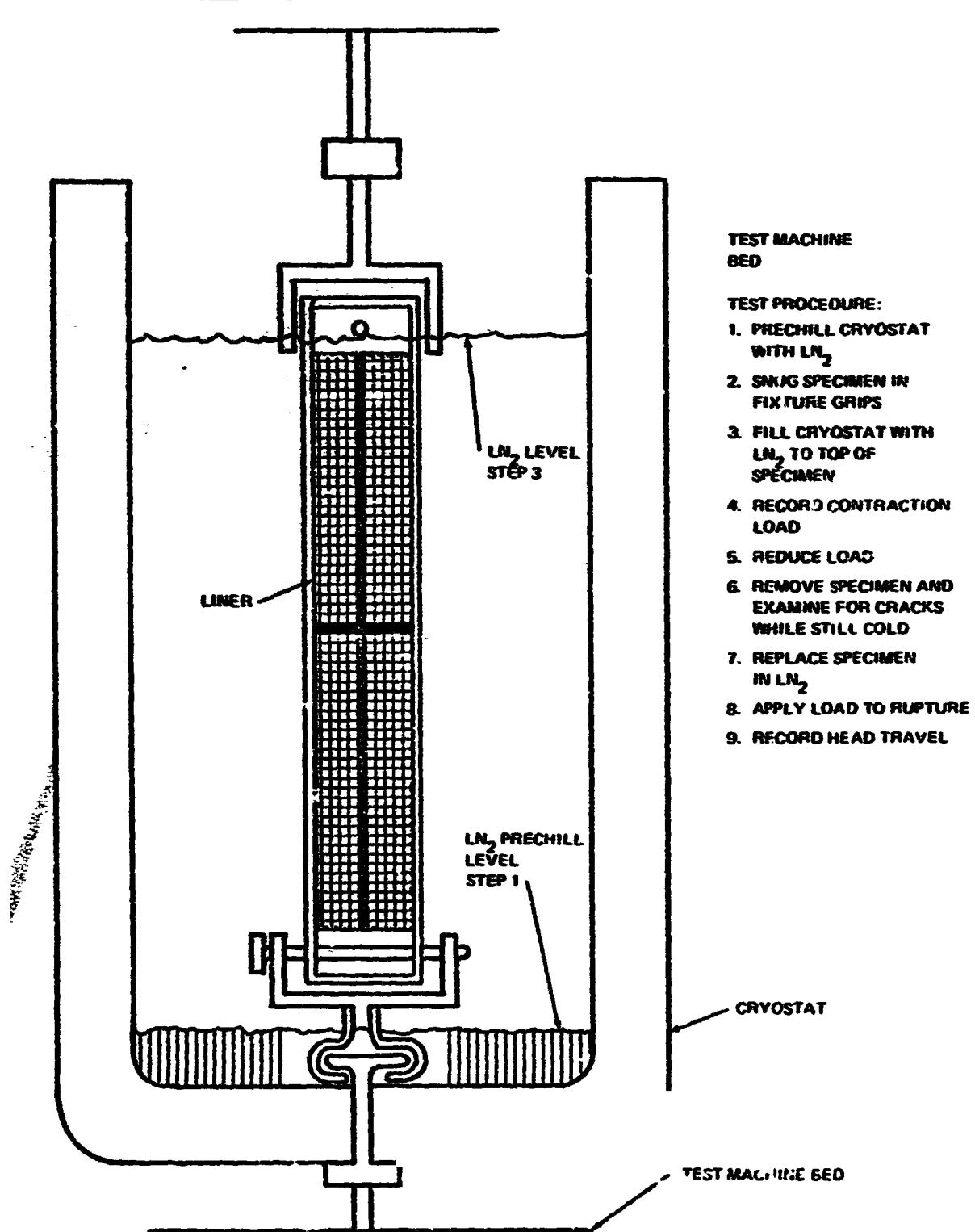


Figure 2-6. Test Setup for Joint Contraction 3D Foam

Table 2-2. LN<sub>2</sub> (-320°F) Contraction Tests on Gap Fillers for 8-D FOAM

Configuration	Gap Filler Material	LN <sub>2</sub> Contraction Load	Rupture Strength in LN <sub>2</sub>
BLOCK BOND RUPTURE 0.64 cm wide GAP FILLER BETWEEN 3D FOAM	POLYURETHANE RESIN U-135/MOCA WITH 1% Z6040 SILANE ADDITIVE FILLED WITH PHENOLIC MICROBALLOONS: 1 PART FILLER 3 PARTS RESIN BY WEIGHT	70 Kg (155 LB); 2 CRACKS IN GAP FILLER VISIBLE AFTER LN <sub>2</sub> EXPOSURE	168 Kg (370 LB); 4 CRACKS VISIBLE IN GAP FILLER AFTER RUPTURE AT BLOCK BOND
BLOCK BOND RUPTURE	EPOXY RESIN EPOXY 828/CL FILLED WITH PHENOLIC MICROBALLOONS 1 PART FILLER 3.6 PARTS RESIN BY WEIGHT	82 Kg (180 LB); 4 CRACKS VISIBLE IN GAP FILLER AFTER LN <sub>2</sub> EXPOSURE	175 Kg (385 LB); 6 CRACKS VISIBLE IN GAP FILLER AFTER LN <sub>2</sub> RUPTURE AT BLOCK BOND
BUTT JOINT RUPTURE	POLYURETHANE RESIN U-135/MOCA WITH 1% Z6040 SILANE ADDITIVE FILLED WITH PHENOLIC MICROBALLOONS 1 PART FILLER 3 PARTS RESIN BY WEIGHT	77 Kg (170 LB); 3 CRACKS VISIBLE IN LONGITUDINAL JOINT	104 Kg (230 LB); RUPTURED AT BUTT JOINT
BUTT JOINT RUPTURE	EPOXY RESIN 828/CL FILLED WITH PHENOLIC MICROBALLOONS; 1 PART FILLER 3.6 PARTS RESIN BY WEIGHT	67 Kg (147 LB); 3 CRACKS VISIBLE IN LONGITUDINAL JOINT	91 Kg (200 LB); RUPTURED AT BUTT JOINT
BUTT JOINT RUPTURE	LEFKOWELD 211A/LZ ADHESIVE	50 Kg (110 LB); MULTIPLE SMALL CRACKS VISIBLE IN LONGITUDINAL JOINT	316 Kg (696 LB); RUPTURED AT BUTT JOINT

- The factor limiting the reduction in density of the joint filler formulation is the achievement of a trowelable mixture that will easily flow into a gap between pieces of 3D foam and remain in position until vacuum bag pressure is applied to the liner. Density formulations of 0.24 or 0.32 g/cm<sup>3</sup> (15 or 20pcf) are powdery and do not pack tightly in the narrow gaps between pieces of 3D foam.

For these reasons, the next series of contraction tests in LN<sub>2</sub> and in LH<sub>2</sub> was confined to the joint filler formulation consisting of:

- 100 parts phenolic microballoons (Union Carbide and Chemical Co.)
- 25 parts 0.64-cm (0.25-in.) milled glass fibers
- 300 parts Epon 828/CL Epoxy resin (Shell Chemical Co.)

This mixture, having a density between 0.40 and 0.48 g/cm<sup>3</sup> (25 and 30 pcf) and a consistency of dough, was injected into the 3D foam joint using an air-pressure operated sealant gun and disposable nozzle. The aluminum blocks were coated with Lefkoweld 211A/LZ adhesive and nested in place at each end of the specimen. The 116 glass fabric liner, 5.1 cm (2 in.) wide, was impregnated with Epon 828/CL resin and wrapped completely around both end blocks and two faces of the 3D foam specimen as shown in Figure 2-5. The assembly was then placed in a vacuum bag for curing all components together for the following cycle:

- 16 hours at 52°C (125°F) under 51-cm (20-in.) Hg pressure
- Remove vacuum bag
- Postcure with no applied pressure
- 1 hr at 75°C (180°F)
- 1 hr at 93°C (200°F)
- 1 hr at 111°C (250°F)
- 32 hr at 149°C (300°F)

Results of contraction tests in LN<sub>2</sub> (-196°C) (-320°F) and LH<sub>2</sub> (-253°C) (-423°F) are presented in Table 2-3.

Table 2-3  
CONTRACTION IN LN<sub>2</sub> AND LH<sub>2</sub>

Preload at 25°C (77°F)	Contraction Load with LN <sub>2</sub> Chill Down	Rupture Load In LN <sub>2</sub>	Contraction Load with LH <sub>2</sub> Chill Down	Rupture Load In LH <sub>2</sub>
1 11.3 kg (25 lb)	134 kg (295 lb)	457 kg (1,005 lb)	125 kg (275 lb)	447 kg (985 lb)
2 4.5 kg (10 lb)	295 kg (200 lb)	454 kg (1,000 lb)	132 kg (290 lb)	395 kg (870 lb)

The significant factors determined by these contraction tests were as follows:

- The filled joint between 3D foam pieces has no deleterious effect on the structural integrity of the glass liner.
- The resins used in (1) the joint filler, (2) the glass liner, and (3) the tank wall adhesive are structurally compatible with each other and have nearly identical working life properties necessary for concurrent bonding operations.
- The load induced by cryogenic contraction with the ends of the specimen restrained is less than one-third of the rupture strength.

The test method and procedure used for these contraction tests allows only the gross effects to be evaluated on the composite specimen. Since the results indicate an ample margin of strength between the measured contraction load and the rupture strength, no serious effort was expended to negate the effects of load-train contraction or to identify the stress associated with each element of the composite specimen.

However, the liner was capable of carrying 53.5 kg per centimeter (300 lb per inch) of width X 10.2 cm (4 in.) = 550 kg (1,200 lb) while the butt joined 3D foam was capable of carrying only 91 kg (200 lb). The composite test resulting in rupture at 410 kg (900 lb) was less than might be predicted but was three times higher than the load developed by thermal contraction.

To evaluate its resistance to high temperature, the gap filler was exposed to 177°C (350°F) for up to 116 hours. The percent weight loss is shown in Figure 2-7 and was sufficiently low that outgassing effects could be safely ignored. (Less than 3 percent after 100 hr exposed to (350°F) 177°C.)

#### 2.4 MINIMUM TANK-WALL ADHESIVE WEIGHT (TASK 4)

The objective of this task was to determine the minimum tolerance on Lefkoweld 211A/LZ adhesive weight that can be used to bond the optimized

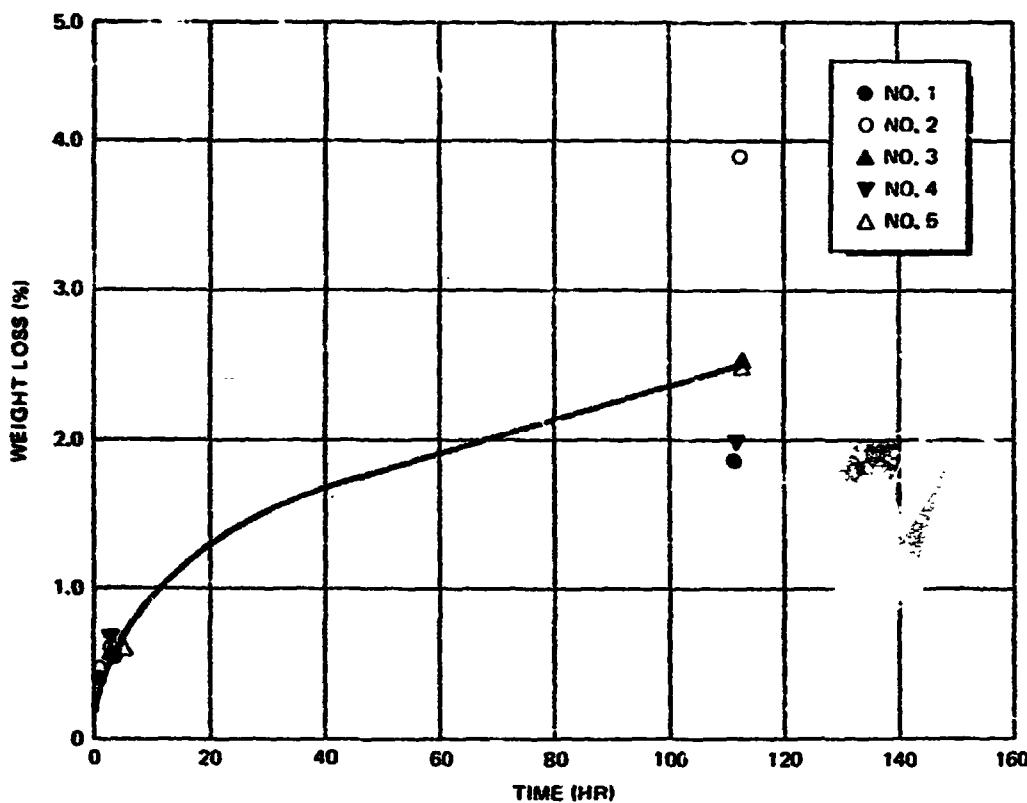


Figure 2-7. Weight Loss of Microballoon Joint Filler at 177°C (350°F)

3D foam to the tank wall reliably using Lefkoweld 211A/LZ adhesive manufactured by Leffingwell Chemical Company, Brea, California

This effort was based on recent experiments in which the entire surface of 3D foam tiles was coated with adhesive, in accordance with the baseline, at 0.48 to 0.54 kg/m<sup>2</sup> (45 to 50 g/ft<sup>2</sup>). Cellophane film was then pressed against the adhesive layer and peeled off, taking the adhesive off the foam surface, but leaving adhesive on the thread ends. Laboratory tests indicated that acceptable bonds could be produced at temperatures from 177°C (350°F) to -196°C (-320°F) with an average adhesive coating weight of only 0.19 kg/m<sup>2</sup> (18 g/ft<sup>2</sup>).

The specific weight of adhesive per unit surface area is not necessarily an indication of the bond strength that can be achieved when considering

adhesive deposits less than  $0.48 \text{ kg/m}^2$  ( $45 \text{ g/ft}^2$ ). The nominal adhesive application of  $0.48 \text{ kg/m}^2$  ( $45 \text{ g/ft}^2$ ) or higher provides a uniform layer over the entire surface of the 3D foam. To achieve adhesive weights less than  $0.48 \text{ kg/m}^2$  ( $45 \text{ g/ft}^2$ ), it is necessary to remove the adhesive on the foam surface between Z thread ends without disturbing the fillet of adhesive locked into the frayed Z threads. The Z-thread fillet contact area to the metal tank wall is estimated at  $0.15 \text{ cm}$  ( $0.06 \text{ in.}$ ) in diameter and the nominal number of Z threads are  $4.38 \text{ per cm}^2$  ( $28.3 \text{ in.}^2$ ).

$$\pi 0.075^2 \cdot 4.38 \text{ threads/cm}^2 = \sim 8.0 \text{ percent of surface area}$$

This calculation would indicate that  $0.04 \text{ kg/m}^2$  ( $3.6 \text{ g/ft}^2$ ) of the  $0.48 \text{ kg/m}^2$  ( $45 \text{ g/ft}^2$ ) adhesive layer (thread fillet area) is actually contributing 90 percent of the bond strength as measured by test. The remaining 10-percent load-carrying ability is provided by the foam bonded to the metal plate which at 2-percent elongation will develop  $0.14 \text{ MN/m}^2$  ( $20 \text{ psi}$ ) as noted by Figure 2-8.

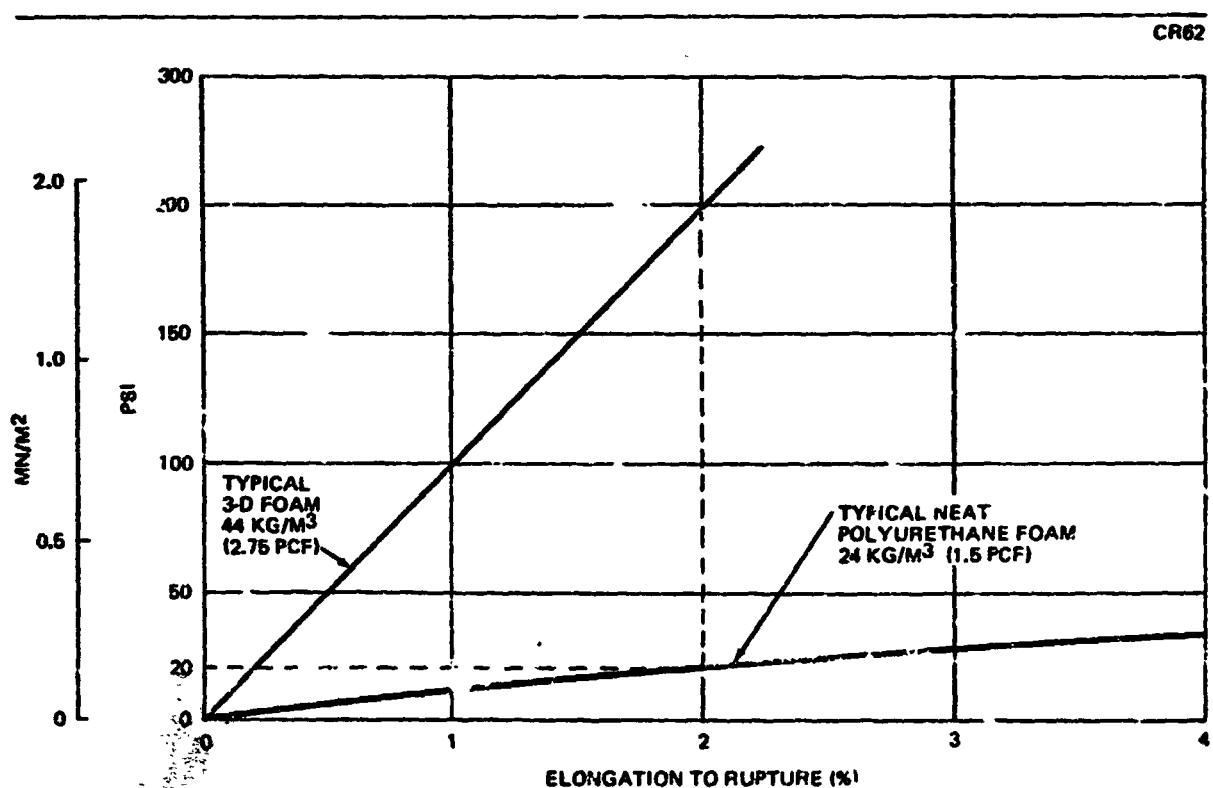


Figure 2-8. Typical Elongation to Rupture

Therefore, the size of the fillet bond to the Z thread end is critical in achieving acceptable overall strength, and the measured weight per unit area of adhesive is of secondary importance. The weight per unit area is still measured but can only be used to indicate the broad range where the highest probability of obtaining acceptable strength was statistically achieved. Visual confirmation of each thread end having a glob of adhesive has provided a better indication of acceptable bond strength than by using the weight measurement alone.

A review of the tensile bond strength tests presented in Table 2-4 showing the bond strength of panels having adhesive weights from  $0.07 \text{ kg/m}^2$  ( $7 \text{ g/ft}^2$ ) to  $0.28 \text{ kg/m}^2$  ( $26 \text{ g/ft}^2$ ), would place the lower limit on adhesive weight at  $0.19 \text{ kg/m}^2$  ( $18 \text{ g/ft}^2$ ). However, many panels having higher adhesive weight were not bonded for strength testing because the thread ends were observed to be almost dry.

To provide greater assurance of the thread end retaining the maximum size glob of adhesive, the following coating operating was established:

- Apply the mixed adhesive to the 3D foam surface, as shown in Figure 2-9, at a temperature of  $49^\circ\text{C}$  ( $120^\circ\text{F}$ ) to  $60^\circ\text{C}$  ( $140^\circ\text{F}$ ) and wipe into the frayed thread ends to achieve maximum penetration.
- Immediately chill the adhesive layer to  $21^\circ\text{C} \pm 2.8^\circ\text{C}$  ( $70^\circ\text{F} \pm 5^\circ\text{F}$ ) to lock the adhesive in place within the frayed thread ends but retain the tack necessary for the roller to remove the excess when performing the next step.
- Remove the adhesive between thread ends by rolling with a felt paint roller covered with 6-mil polyethylene film as shown in Figure 2-9. Scrape off the excess adhesive that will collect on the

**Table 2-4**  
**LOW WEIGHT 211A/62 ADHESIVE TENSILE  
 BOND STRENGTH**

Adhesive Coating Weight $\text{kg/m}^2$	(g/ $\text{ft}^2$ )	Bond Strength 196°C (-330°F)		Bond Strength 25°C (77°F)		Temperature at Rupture Under Stress of 0.7 MN/m $^2$	
		MN/m $^2$	(psi)	MN/m $^2$	(psi)	°C	(°F)
0.484	(45)	1.43	(208; Average)		(225) Average	>177	>(350)
0.280	(26)					143 160 172 175	(290) (320) (342) (348)
0.270	(25)						
0.237	(22)					173 154 151 165 143 171	(346) (310) (322) (330) (290) (340)
0.194	(18)	1.41 1.45 1.62	(204) (210) (235)			171 168 168	(340) (335) (245)
0.172	(16)			0.56 0.88 0.73 0.66	(81) (128) (106) (95)	160 165	(320) (330)
0.108	(10)			0.43 0.52 0.61 0.59 0.42 0.49 0.62	(63) (75) (88) (85) (61) (71) (90)		Not able to start heat cycle. These specimens ruptured at ambient temperature.
0.075	(7)	1.14 0.59 1.21 0.67	(166) (96) (176) (124)	0.54 0.54 0.69	(78) (78) (100)		

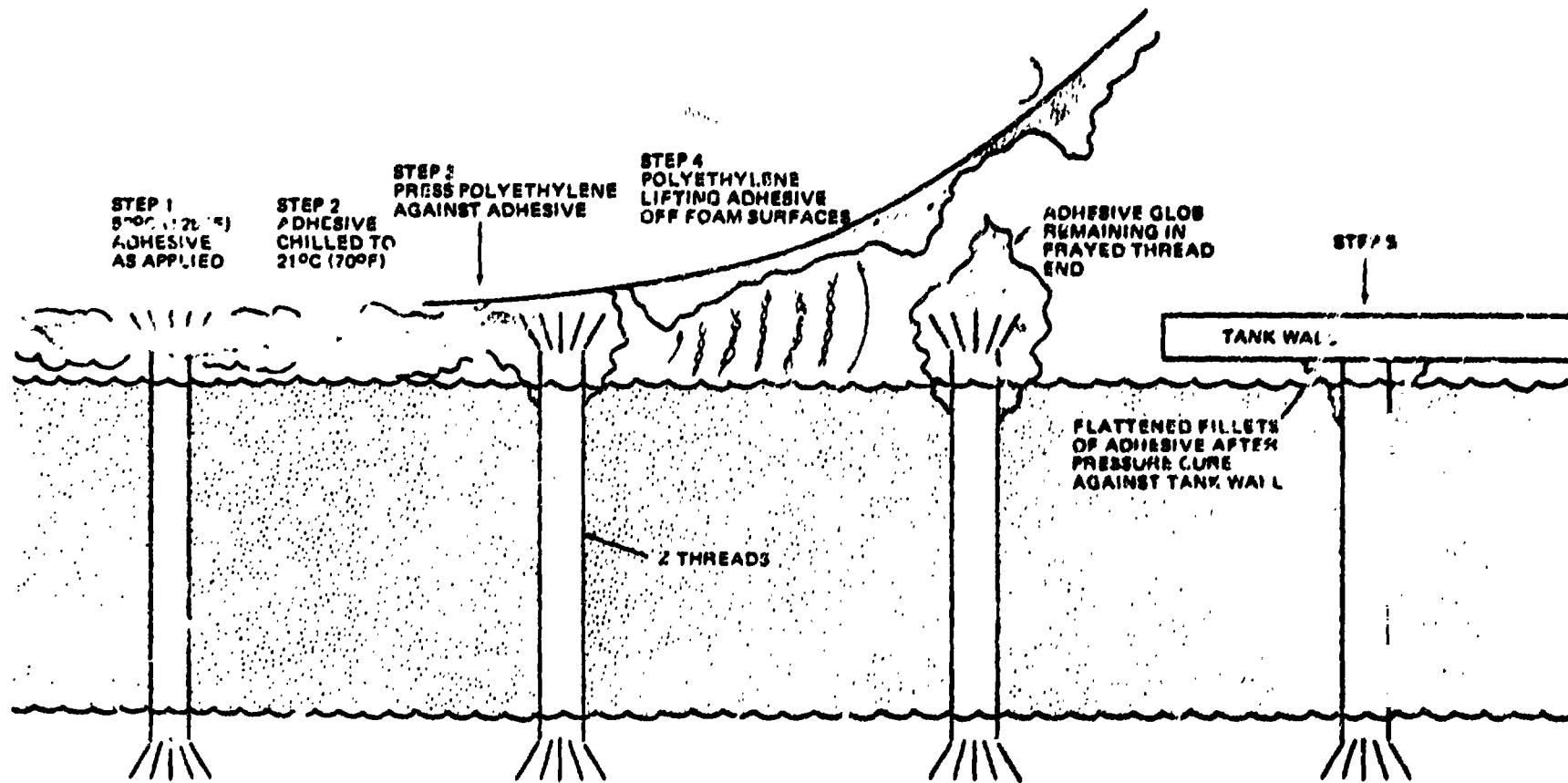


Figure 2-9. Steps to Application of Adhesive on Thread Ends

roller but leave the high-tack surface condition. After making contact with the adhesive coating on the 3D foam, the 5-cm (3-in.) diameter roller should exert a lifting action, as opposed to peeling action, to draw the adhesive vertically away from the foam surface and leave a long string of adhesive attached around the Z thread end. Observe in Figure 2-9 that each thread end retains a glob of adhesive, while leaving the foam relatively clean except for random strings of adhesive.

- Place the adhesive "coated" panel against the tank wall and apply vacuum bag pressure for the normal cure cycle used for Lefkoweld 211A/LZ.

The test panels prepared in this fashion retained a visual glob of adhesive on each thread end and the adhesive weighed between 0.23 and 0.29 kg/m<sup>2</sup> (22 and 27 g/ft<sup>2</sup>). Continued rolling with the polyethylene coated roller will remove more of the adhesive on the foam surface but will also reduce the fillet adhesive on the thread ends.

In summary, the results of this program to evaluate the minimum tolerable weight to bond Bx-251A-3D to aluminum indicated that a value of 0.28 kg/m<sup>2</sup> (26 g/ft<sup>2</sup>) was the minimum reliable weight.

## 2.5 SYSTEM EVALUATION TESTS (TASK 5)

There were two objectives for this task, the first was to demonstrate the ability of the baseline system, optimized under Phases II and III, to sustain simulated Shuttle tanking, pressurization, and reentry heating cyclic stresses in the 1-m (3-ft) dome test. The second objective was to determine the ability of a lightweight system, optimized under Phase III, to pass a similar 1-m (3-ft) dome test. Two tests were run with 7 LH<sub>2</sub> cycles each.

At the conclusion of Phase I, a 1-m (3-ft) dome plate was insulated. The dome was visually, sonically, and ultrasonically inspected prior to testing.

The dome and insulation were then subjected to 25 thermal cycles and again inspected. Tensile plug tests were conducted on the liner and the tank wall bond. This test provided a basis for close comparison with the results to be obtained during Phase III.

This task required the fabrication of two new dome plates made of 2219-T87 aluminum alloy. These 0.64 x 122 x 122-cm (0.25 x 48 x 48-in.) sheets were contoured by spinning to the 204-cm (80-in.) spherical radius. The outside surface of the dome plates were then chemically milled to obtain a wall thickness of 0.011 cm (0.045 in.) in the spherical area leaving a 0.64-cm (0.25-in.) thick flange. The surfaces of the dome plates to receive bonded 3D foam insulation were anodized to represent the Shuttle tank surface preparation.

The insulation configuration depicted in Figure 2-10 used on the first 1-m (3-ft) dome was reviewed by the NASA-COR and consisted of the following materials and bonding operations:

- A. Lefkoweld 211A/LZ adhesive,  $0.48 \text{ kg/m}^2$  ( $45 \text{ g/ft}^2$ ), was deposited on the 3D foam, using the automatic mixing and dispensing machine described in Phase II Summary Report. The coated 3D foam and dome plate were held at  $13^\circ\text{C}$  ( $55^\circ\text{F}$ ) for 30 hours prior to application of vacuum bag pressure to simulate production bonding operations where long layup periods may be required.  
This holding time and temperature conforms to the working life for L211/LZ graph presented in Figure 19 of Reference 4.
- B. BX-251A-3D foam,  $44\text{-kg/m}^3$  (2.75-pcf) density, was used.
- C. Glass fabric 116 was impregnated with epoxy resin, Epon 828/CL, to achieve 50-percent resin content. This liner was handled using simulated production techniques and including a layup time, prior to application of vacuum bag pressure, of 30 hours at  $13^\circ\text{C}$  ( $55^\circ\text{F}$ ).

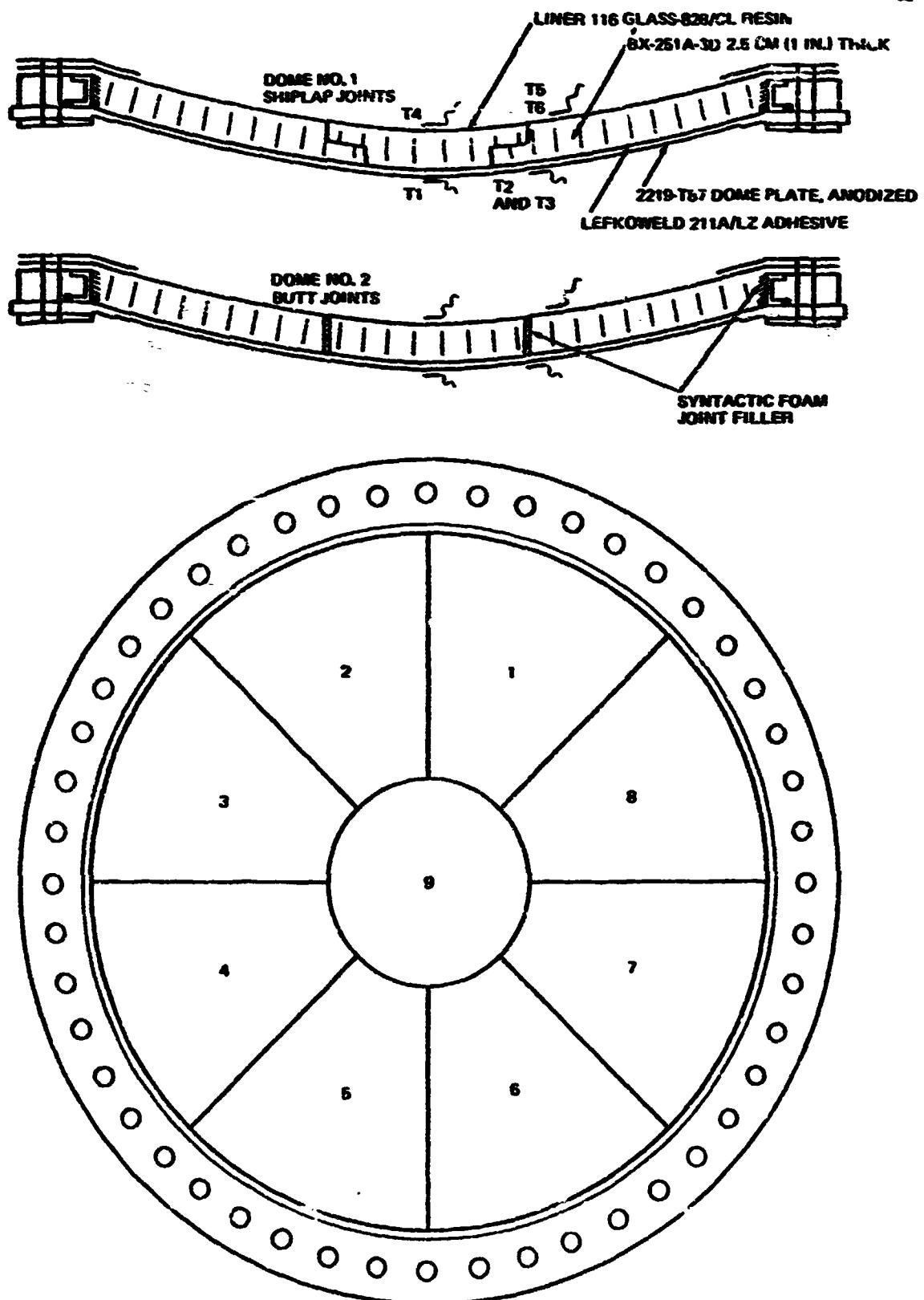


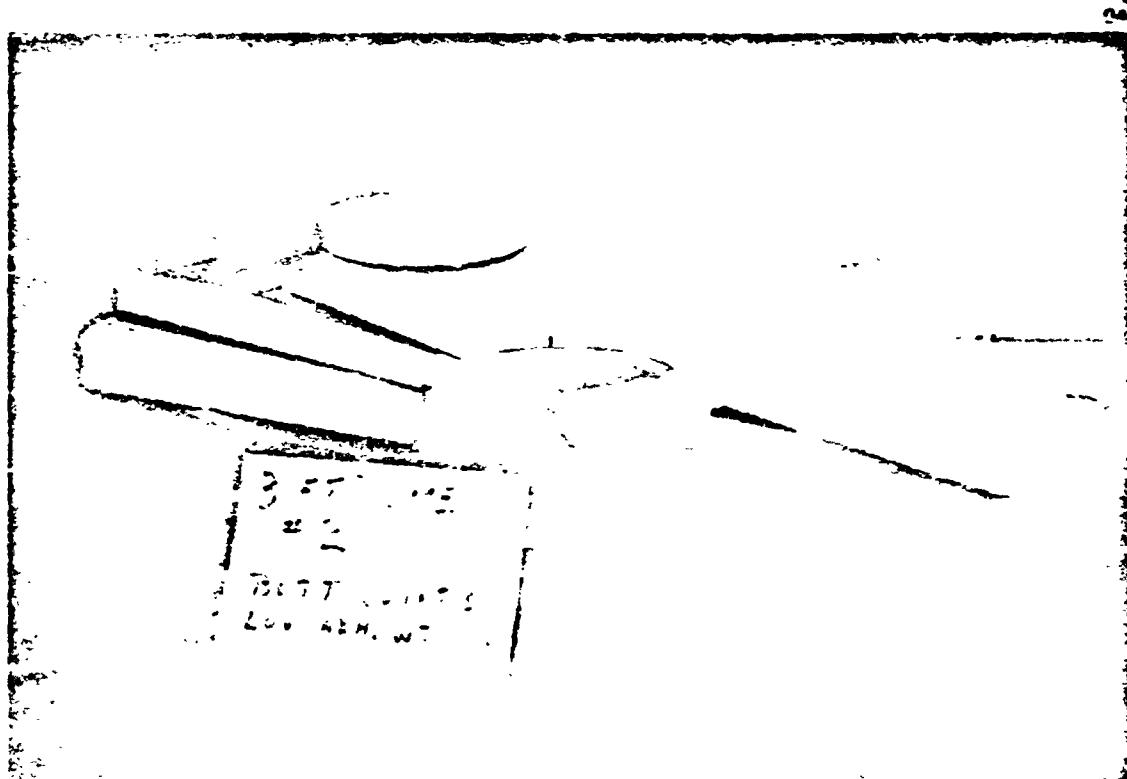
Figure 2-10 1m (3 ft) Dome No. 1 - Shiplap Joints

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- D. The 3D foam tank-wall bond and the liner-to-3D foam bond was accomplished simultaneously using the same vacuum bag and cure cycle as expected during production operations.
- E. The quality assurance tests described in the Phase II Summary Report (Reference 4), including hardness tests, flow tests, tensile bond strength tests, and debond detection examinations, were performed during and after the bonding operation.

The insulation configuration used in Dome No. 2, shown in Figure 2-10 and Figure 2-11, was fabricated using the same materials used in Dome No. 1. The fabrication details of butt joining the 3D foam and the lightweight adhesive application on the 3D foam are described as follows:

- The mixed adhesive (211A/LZ) was applied mechanically to achieve a weight of  $0.48 \text{ kg/m}^2$  ( $45 \text{ g/ft}^2$ ) on the 3D foam surface while the adhesive was warm,  $52$  to  $57^\circ\text{C}$  ( $125$  to  $135^\circ\text{F}$ ). A polyethylene blade was used to press this adhesive into the frayed Z thread ends.



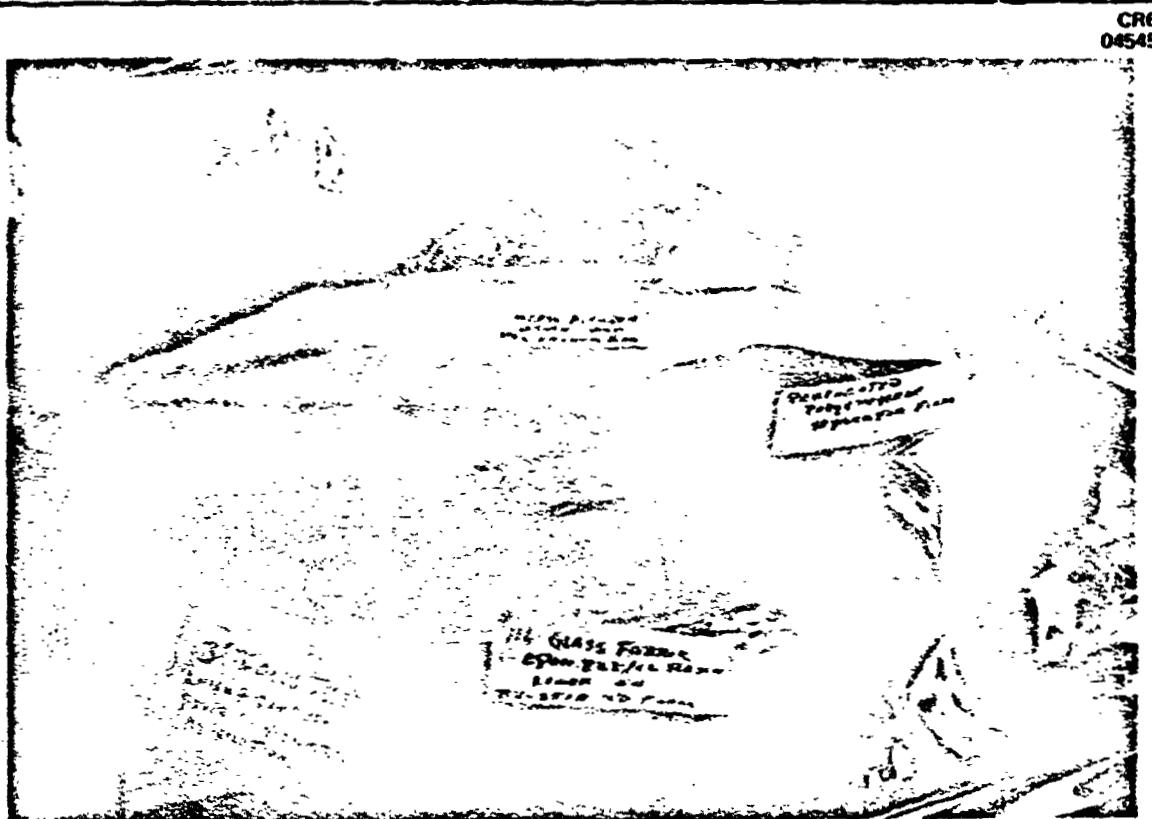
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Figure 2-11. Dome No. 2

- The adhesive layer was cooled to  $21^{\circ}\text{C} \pm 2, 8^{\circ}\text{C}$  ( $70^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ). An 8-cm (3-in.) diameter felt paint roller covered with 5-mil polyethylene film was pressed against the adhesive layer and rolled to remove the adhesive between the Z thread ends. Adhesive globs were visually observed at each Z thread end. The weight of adhesive on each piece of 3D foam was measured and varied between 0.215 and 0.30 kg/m<sup>2</sup> (20 and 28 g/ft<sup>2</sup>).

Q. C. Item: Weight per unit area of adhesive and visual confirmation of adhesive glob at each Z thread end. Adhesive hardness specimens prepared. Tensile bond strength panels prepared on 3D foam on 0.20-cm (0.080-in.) 2219-T87 anodized aluminum plates.

- The coated 3D foam was positioned on the dome-plate surface and the butt-joined edges filled with syntactic gap filler formulated as follows:
  - 300 parts Epon 828/CL resin
  - 100 parts phenolic microballoons
  - 25 parts 0.64-cm (0.25-in.) milled glass fibersThe density of this mixture measured between 0.40 and 0.48 g/cm<sup>3</sup> (25 and 30pcf).
- The 116 glass fabric was impregnated with Epon 828/CL epoxy resin to achieve a resin content of 60 percent by weight as measured on the fabric prior to positioning in place on top of the 3D foam surface.
  - Weight of 116 glass fabric = 0.018 kg/m<sup>2</sup> (10 g/ft<sup>2</sup>)
  - Weight of Epon 838/CL resin = 0.16 ± 0.02 kg/m<sup>2</sup> (15 ± 2 g/ft<sup>2</sup>)
  - 3-mil perforated polyethylene was used as the carrier for the impregnated glass liner and remained adjacent to the liner through the initial cure cycle to serve as a separator film under the dry glass bleeder fabric and polyvinyl chloride (PVC) vacuum bag. This assembly is shown in Figure 2-12.



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Figure 2-12. Dome No. 2 Fabrication Details

**Q. C. Item:** Hardness specimens prepared with Epon 828/CL resin. Tensile bond strength panels prepared with liner on 2.5 x 30 x 30 cm (1x12x12 in.) 3D foam shown in Figure 2-13.

- The assembly was held at 13°C (55°F) for 30 hours prior to application of vacuum pressure to simulate production bonding operations where long layup periods may be required. This holding time and temperature conforms to the working-life graph presented in Figure 19 of Reference 4 for Lefkoweld 211A/LZ adhesive. The working life of the Epon 828/CL impregnated liner is slightly longer at the low holding temperature. However, since both bond lines are pressured and cured simultaneously, the shorter working life of the Lefkoweld 211A/LZ must govern the operational time.

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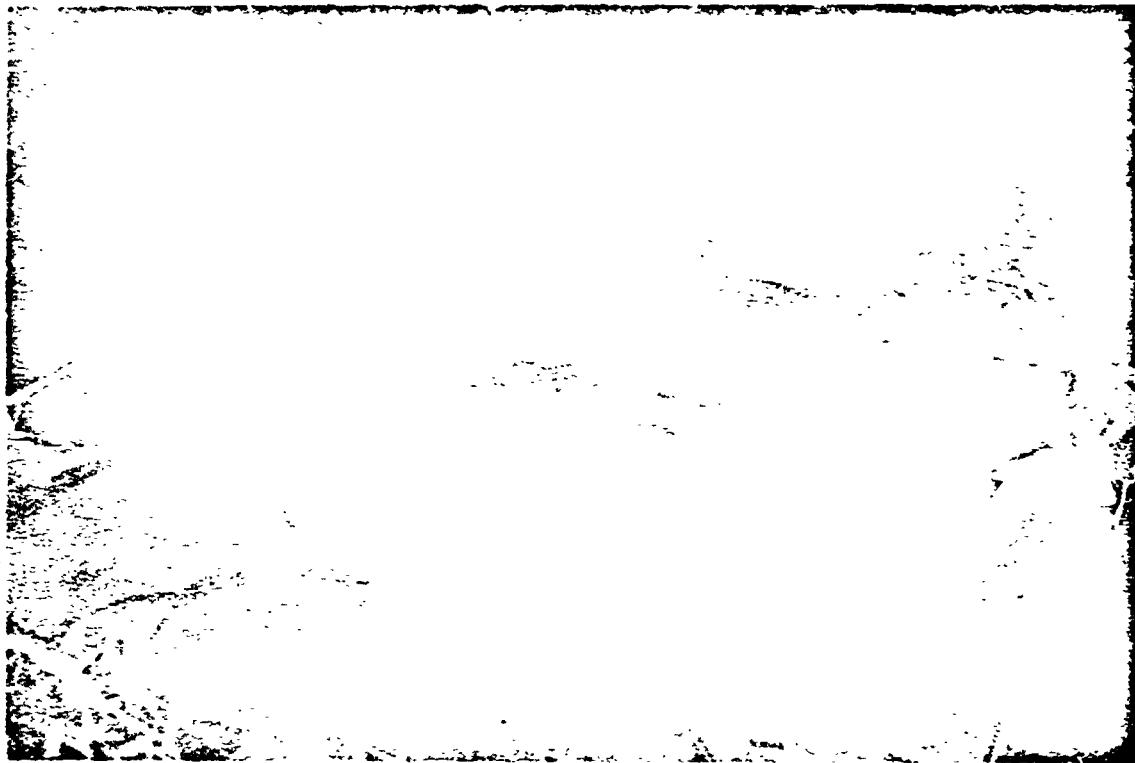


Figure 2-13. Quality Control Coupons

Q. C. Item: Acrylic plate with dry glass threads placed under one corner of the strength test panel just prior to pressure application to give visual confirmation of resin flow during cure cycle.

- Vacuum pressure of 56 cm (22 in.) Hg was applied to the bag while the assembly was at 13°C (55°F). Then the temperature was increased to 52 to 54°C (125 to 130°F) over a period of two hours. This pressure was maintained during the initial cure cycle of 16 hours at 52 to 54°C (125 to 130°F). All the strength test panels were placed in the same PVC bag as used around the 1-m (3-ft) dome.
- The vacuum bag, bleeder fabric, and polyethylene separator film were then removed.

Q. C. Items: Sonic brush examination for liner-to-3D foam debonds. Confirmation of Epon 828/CL resin flow into dry glass threads on acrylic plate.

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- A freshly mixed batch of Epon 282/CL resin was rubbed into the surface of the liner then wiped off leaving resin only in the pores of the liner.
- The postcure cycle was accomplished in accordance with the following schedule:
  - 1 hr at 65°C (150°F)
  - 1 hr at 93°C (180°F)
  - 1 hr at 93°C (200°F)
  - 1 hr at 111°C (250°F)
  - 32 hr at 149°C (300°F)

Q. C. Items: Sonic brush examination for liner-to-3D foam debonds.

Ultrasonic, pulse echo, examination for 3D foam-to-tank wall debonds.

Tensile bond strength tests at -196°C (-320°F) and 177°C (350°F).

Both of these domes are illustrated in Figure 2-14 after all processing steps were completed.

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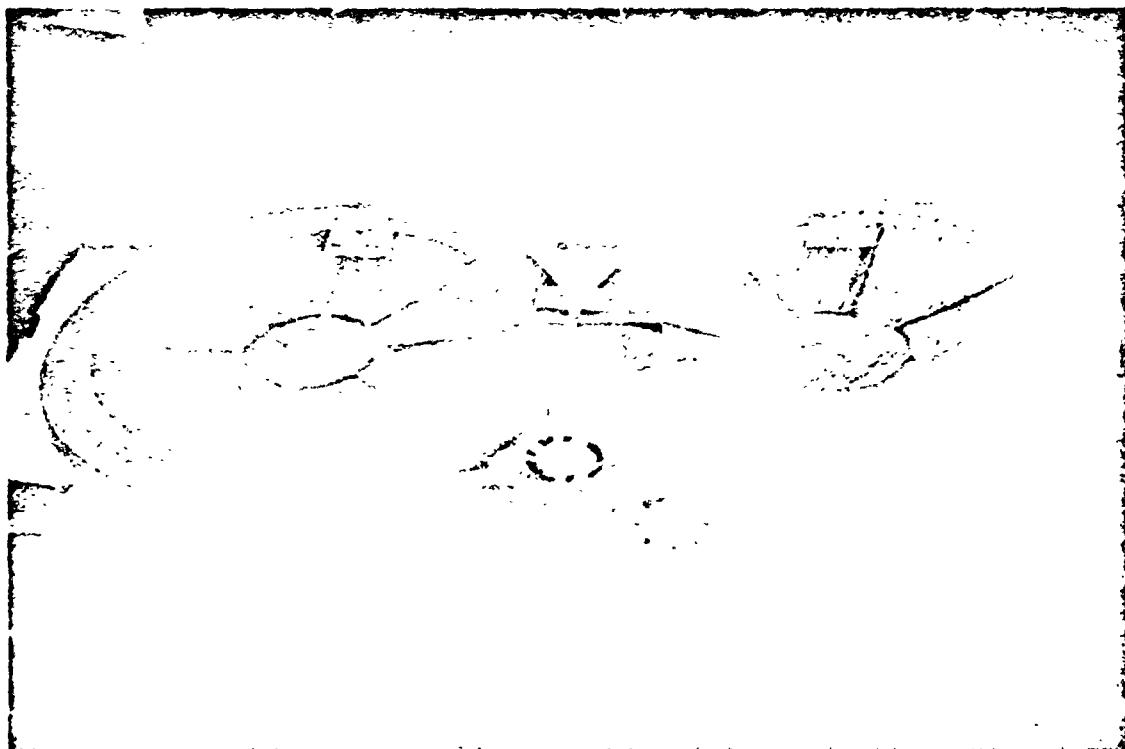


Figure 2-14. Phase III 1 Meter (3 Ft) Domes

### 2.5.1 LH<sub>2</sub> Dome Testing

Seven tests were conducted on each of the two 1-m (3-ft) domes. Each specimen was subjected to repeated cycles of LH<sub>2</sub> fill, chilldown, pressurization, and simultaneous vent and ascent heating. (For a description of the test facility see Reference 1). The primary purpose of the test series was to demonstrate the structural integrity of the two insulation systems - the standard production composite and the lightweight composite.

The specimens were instrumented as follows (see Figures 2-15 and 2-16):

- A. T1, T2, and T3 - thermocouples located on the lower, aluminum side of the test specimen.
- B. T4, T5, and T6 - thermocouples located on the outer surface of the glass liner on the top, or insulated side, of the test specimen.
- C. L1 and L2 - temperature probes located midway and on top of upper dome to indicate 45-percent and 100-percent full.
- D. P1 and P2 - pressure transducers.

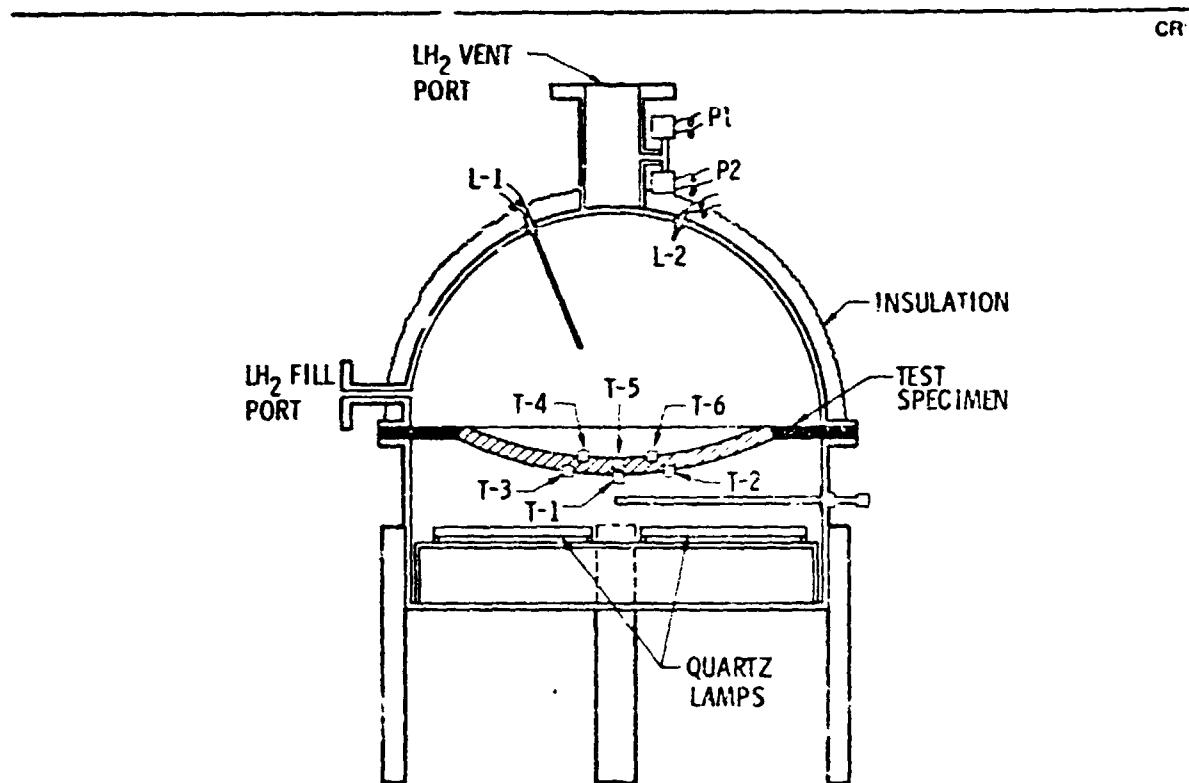


Figure 2-15. Test Specimen and Instrumentation Pickup Points

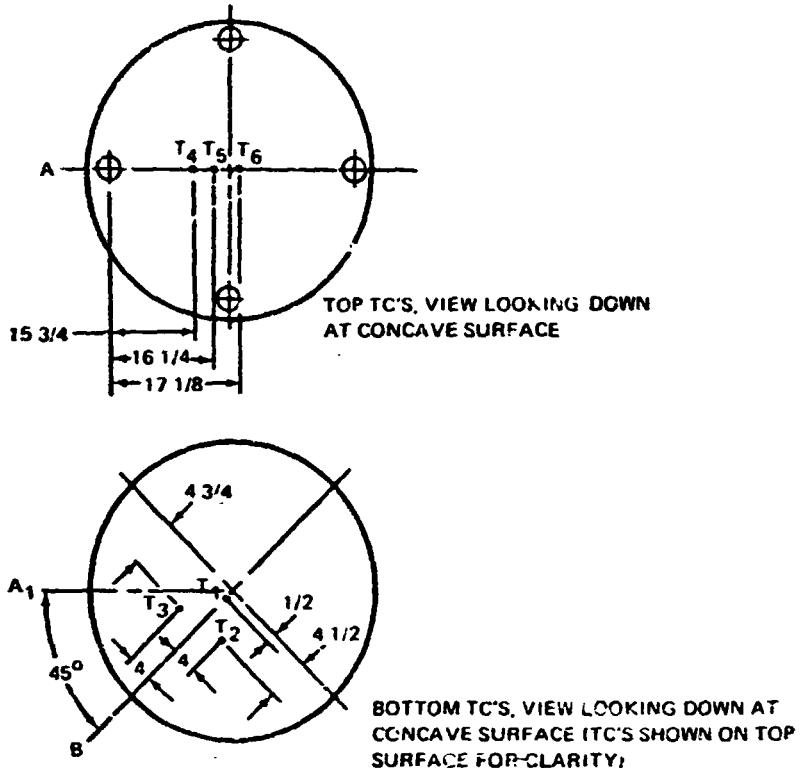


Figure 2-16. Three-Ft Dome Insulation Specimen Thermocouple Location

With the exception of Cycle No. 1, the tests were all similar. Liquid hydrogen was loaded into the top of the test fixture until T3 (dome wall) indicated approximately  $-129^{\circ}\text{C}$  ( $-200^{\circ}\text{F}$ ). The upper sphere of the test fixture was pressurized to  $0.31 \pm 0.01 \text{ MN/m}^2$  a ( $45 \pm 1 \text{ psia}$ ) and held for 5 minutes. At the end of the 5-minute hold, the pressure was vented to ambient and  $\text{LH}_2$  flow stopped. In cycle No. 1 the test was terminated at this point, the specimen allowed to warm to room temperature, and inspected. In all other subsequent cycles, heat was applied on venting. The lower surface of the dome was heated at a uniform rate of temperature increase over a 1,600-second period until T1 indicated  $177^{\circ}\text{C}$  ( $350^{\circ}\text{F}$ ). This temperature was held for 10 minutes, the lamps turned-off, and the test cycle terminated by allowing the specimen to cool to room temperature. This test cycle was essentially identical to that used in Phase I.

### 2.5.2 Discussion of Results

Figures 2-17 through 2-23 summarize the temperature histories obtained during the tests on the No. 1, or standard production quality, dome.

The first cycle was unable to be carried on to completion because of leakage which occurred during the pressurization sequence. The time required for the dome wall to cool to  $-129^{\circ}\text{C}$  ( $-200^{\circ}\text{F}$ ) steadily decreased from 170 minutes for cycle No. 1 to 110 minutes for cycle No. 2 and stabilized at approximately 60 minutes after cycle No. 4. After pressurization and during the 30-minute pressure hold, the temperature during cycles No's 3 through 7 held at approximately  $-150^{\circ}\text{C}$  ( $-238^{\circ}\text{F}$ ).

Figures 2-24 through 2-30 show the time-temperature curves obtained during the tests on the No. 2 dome with the butt-joint tile configuration. These curves are similar with those for dome No. 1, although the curves are displaced approximately 15 minutes to the left (less time).

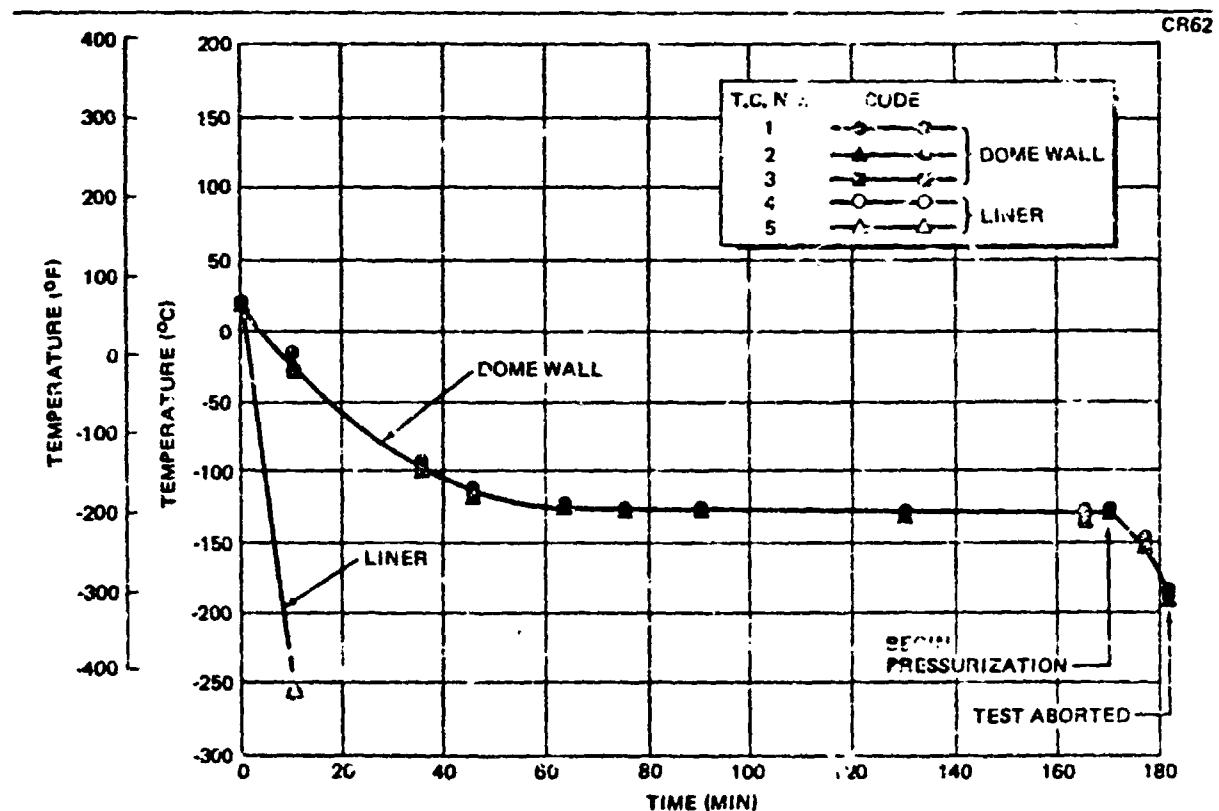


Figure 2-17. 1 m (3 ft) Dome Test -- Shiplap Joints -- Cycle No. 1

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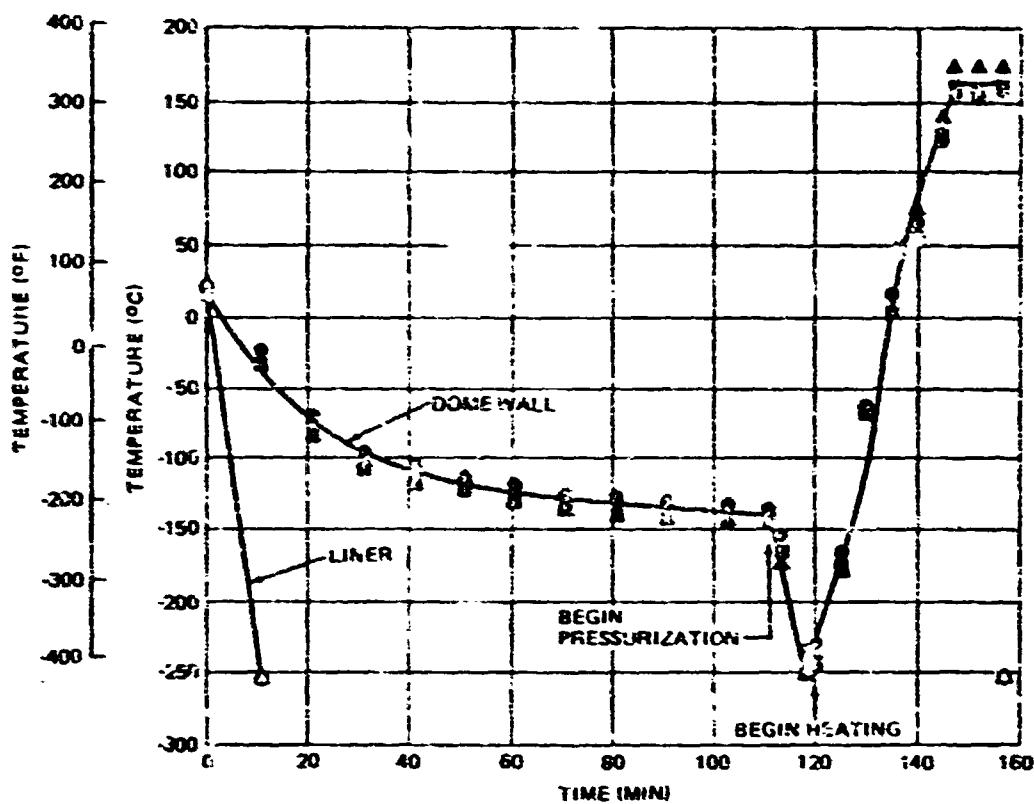


Figure 2-13. 1m (3 ft) Dome Test - Shiplap Joints - Cycle No. 2

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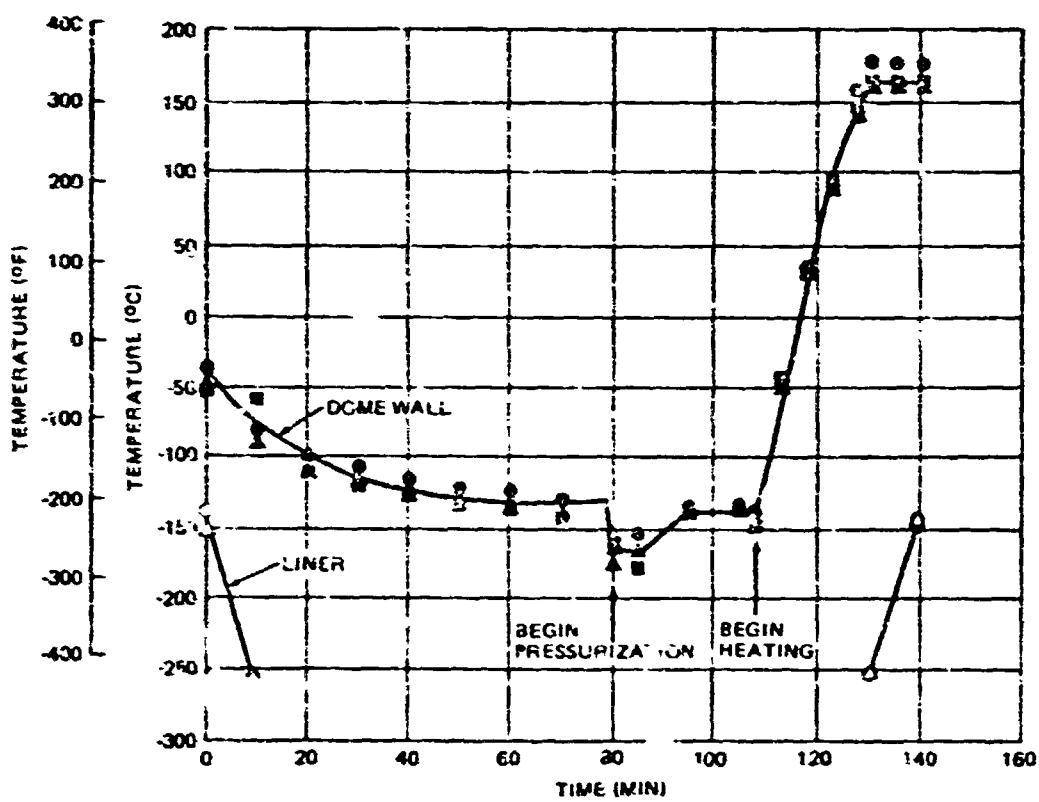


Figure 2-15. 1m (3 ft) Dome Test - Shiplap Joints - Cycle No. 3

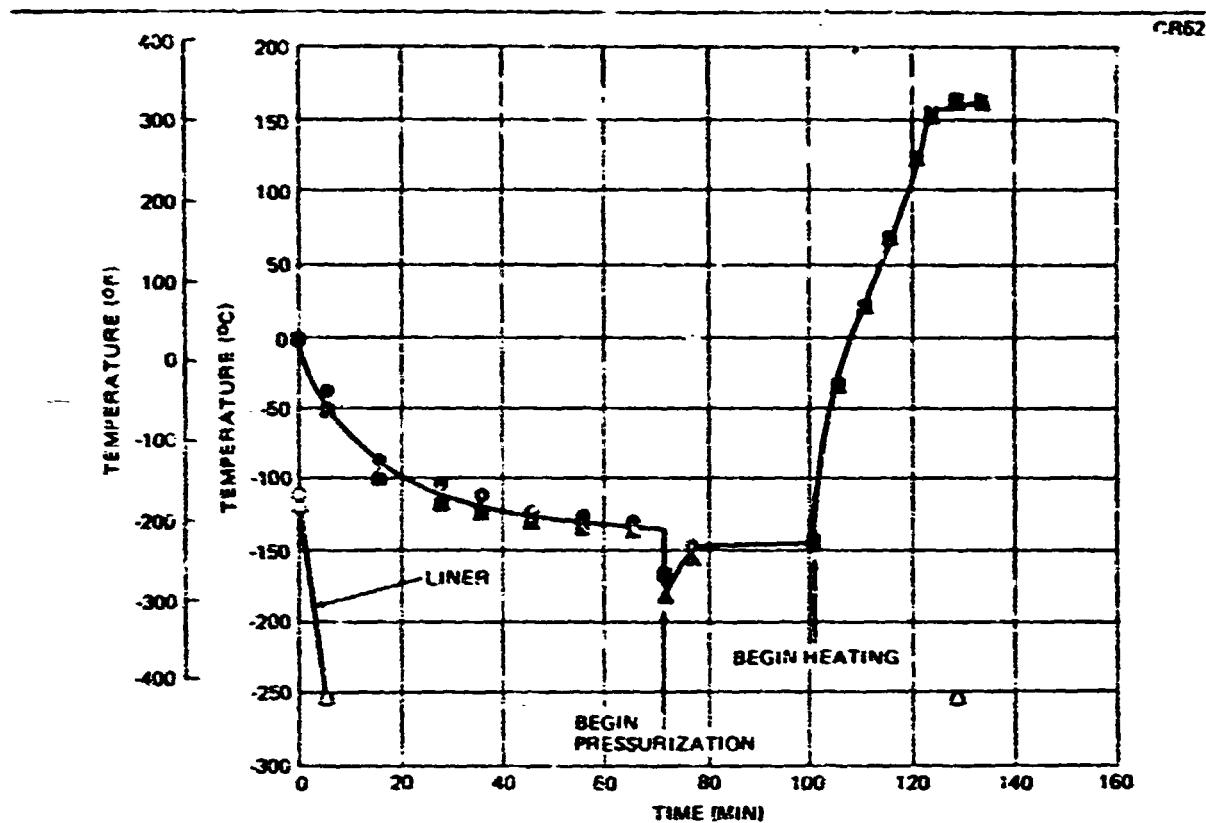


Figure 2-20. 1m (3 ft) Dome Test - Shiplap Joints - Cycle No. 4

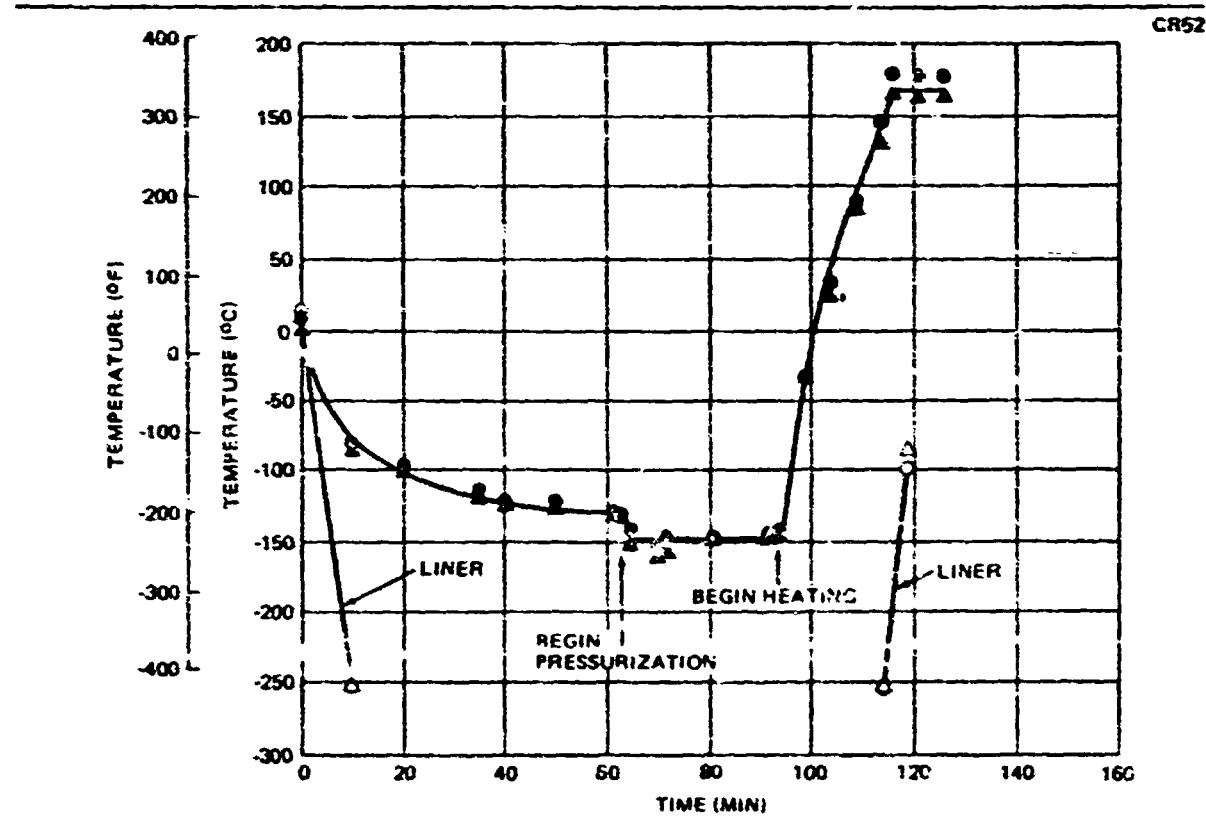


Figure 2-21. 1m (3 ft) Dome Test - Shiplap Joints - Cycle No. 5

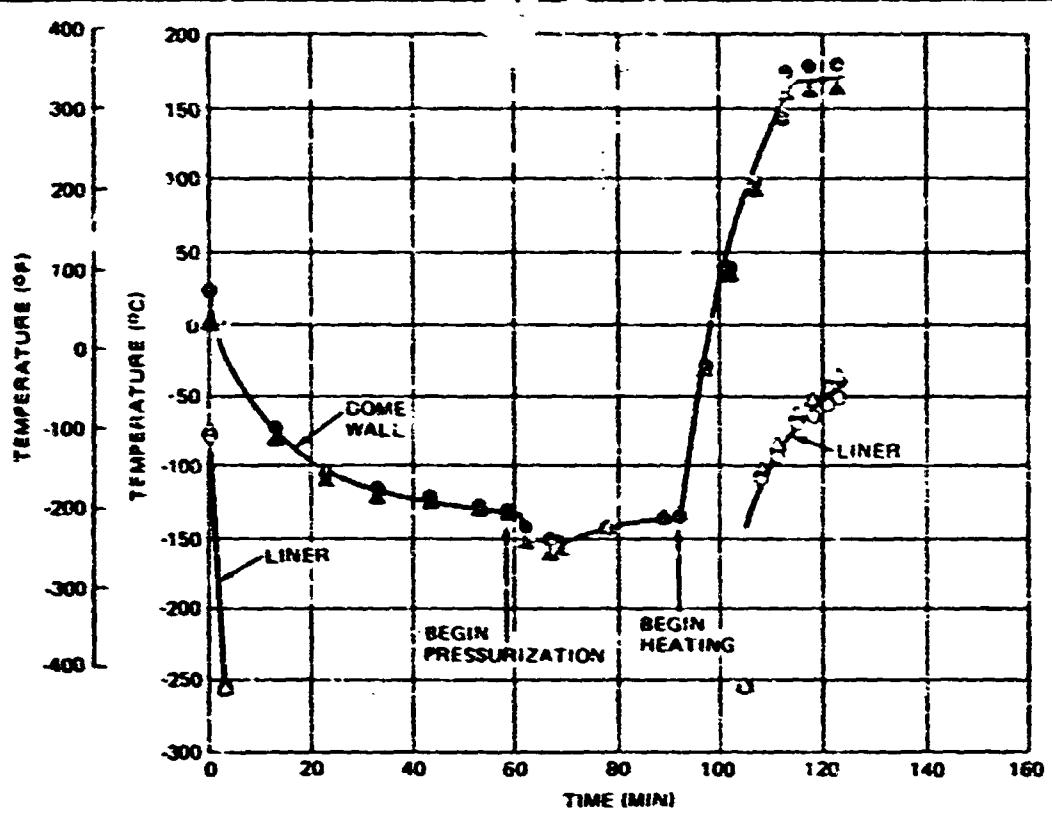


Figure 2-22. 1m (3 ft) Dome Test - Shiplap Joints - Cycle No. 6

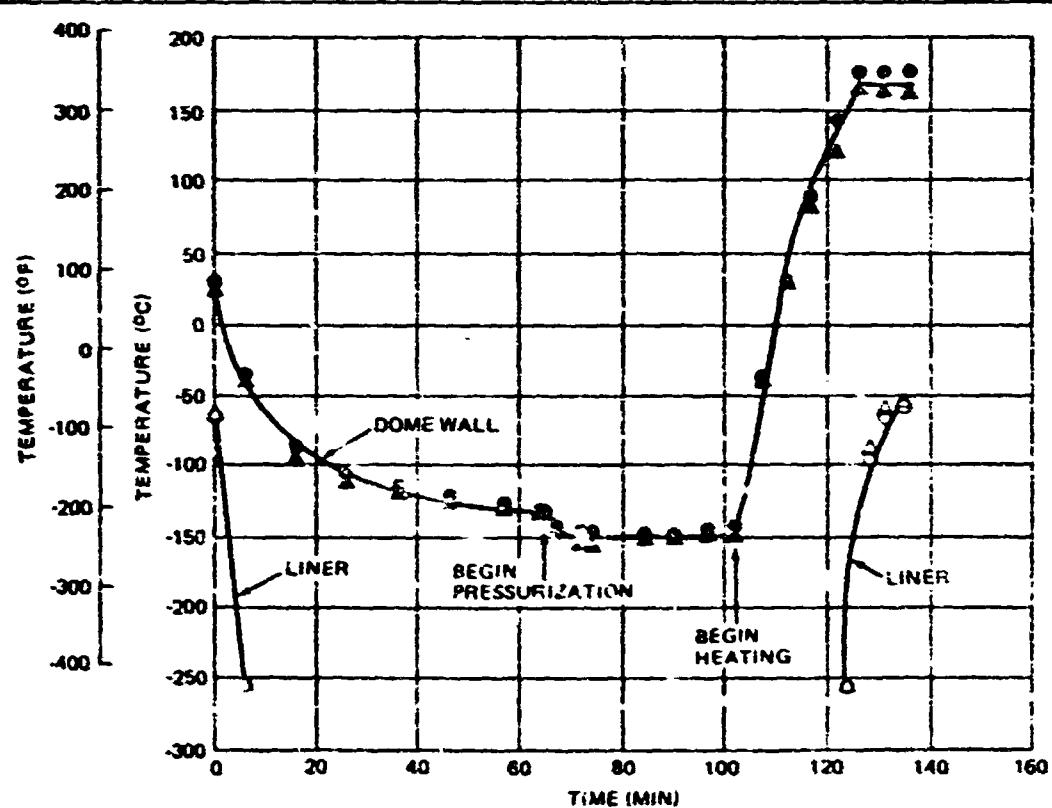


Figure 2-23. 1m (3 ft) Dome Test - Shiplap Joints - Cycle No. 7

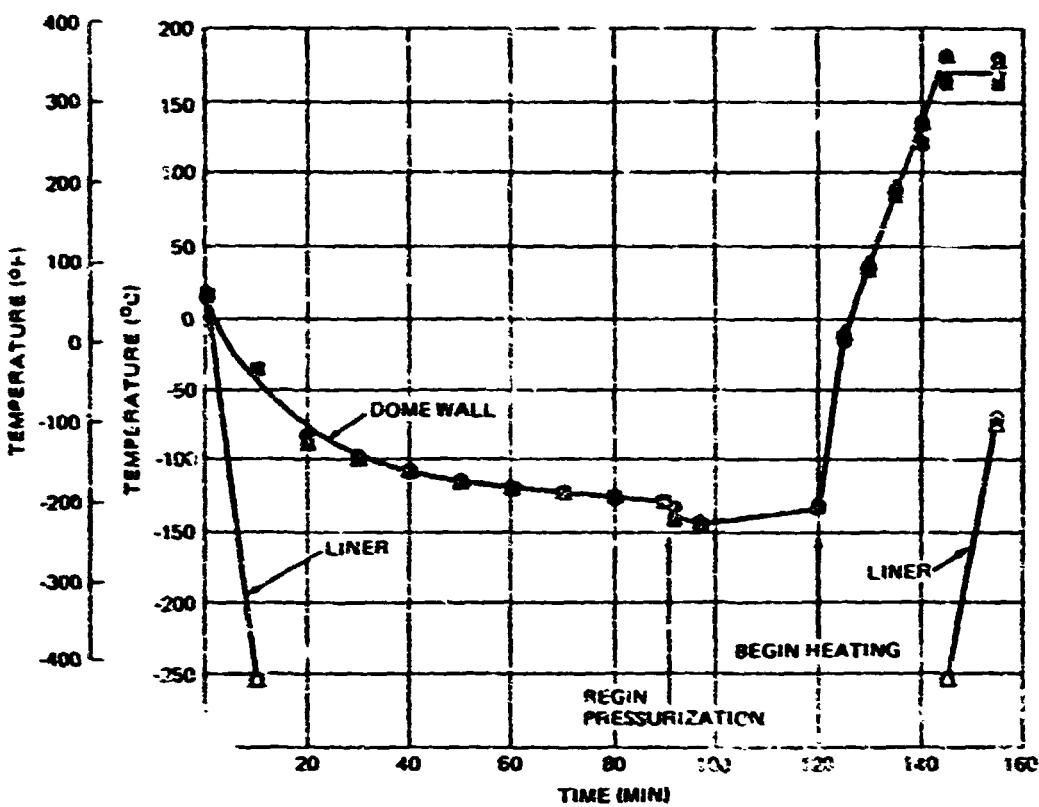


Figure 2-24. 1m (3 ft) Joint Test - Butt Joints - Cycle No. 1

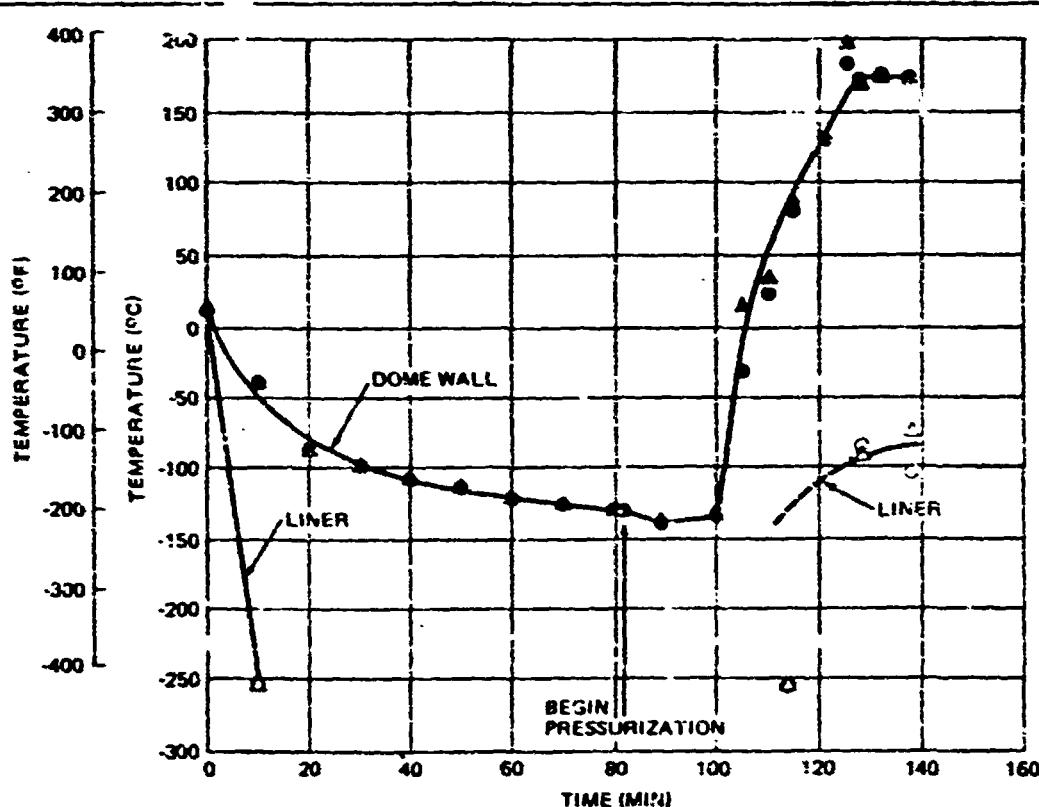


Figure 2-25. 1m (3 ft) Dome Test - Butt Joints - Cycle No. 2

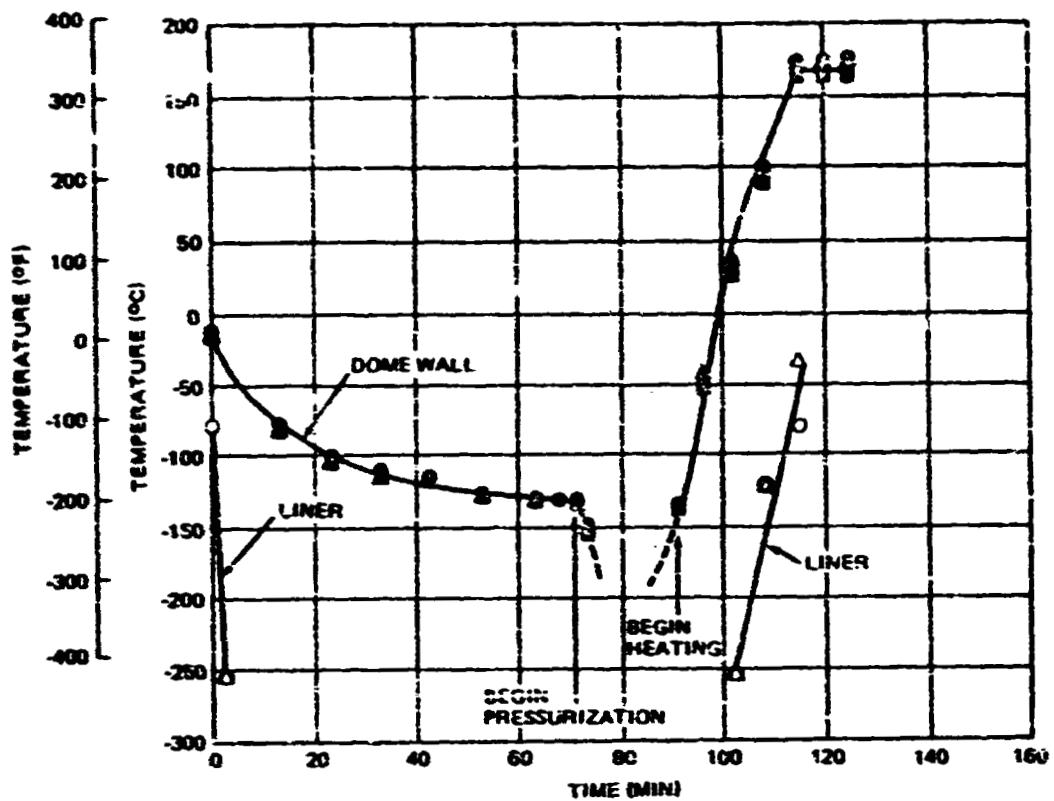


Figure 2-26. 1m (3 ft) Dome Test - Butt Joints - Cycle No. 3

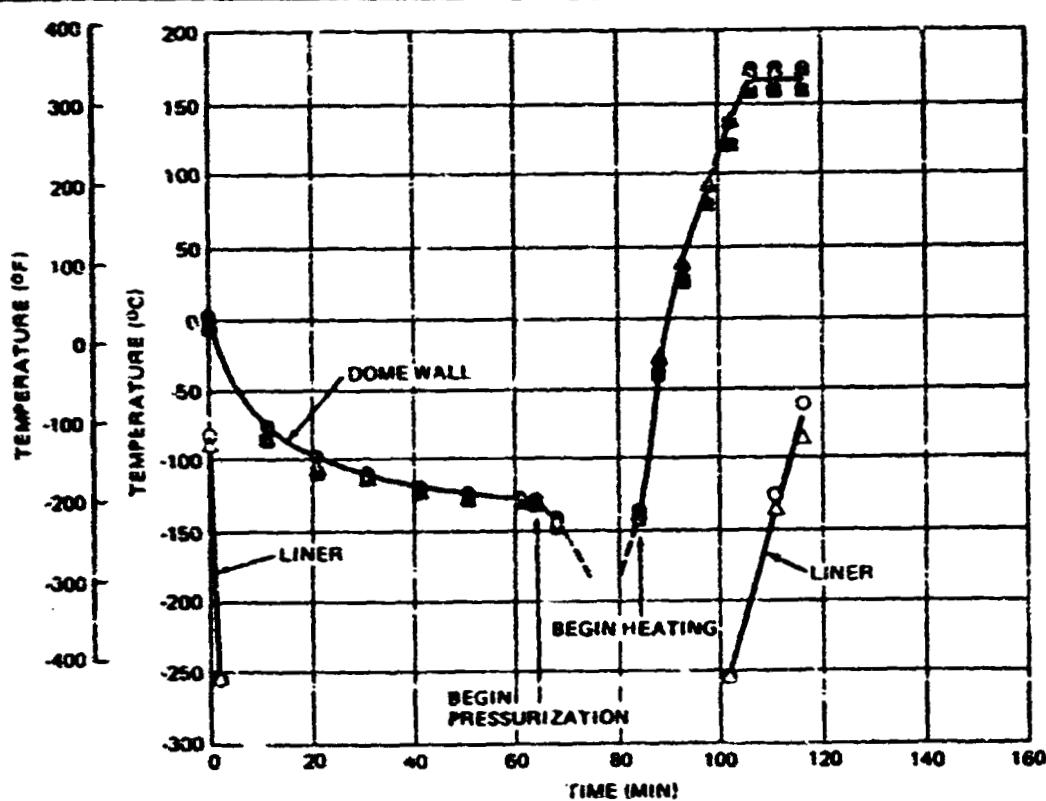


Figure 2-27. 1m (3 ft) Dome Test - Butt Joints - Cycle No. 4

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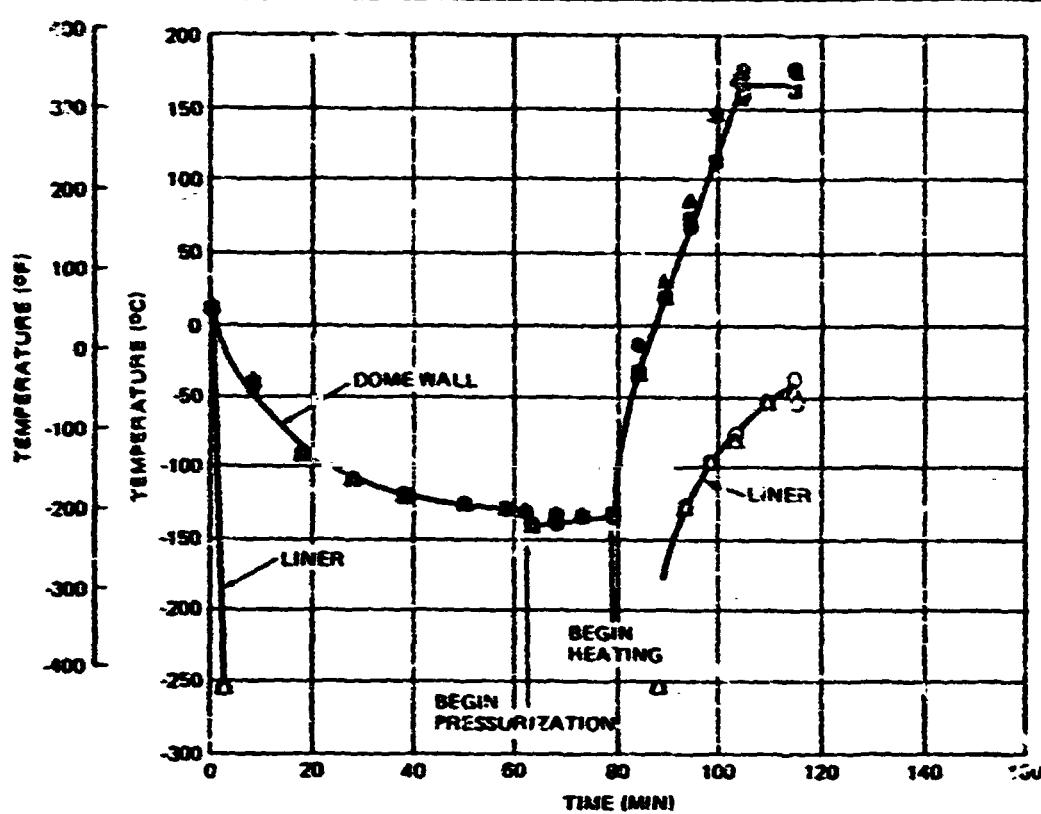


Figure 2-28. 1m (3 ft) Dome Test - Butt Joints - Cycle No. 5

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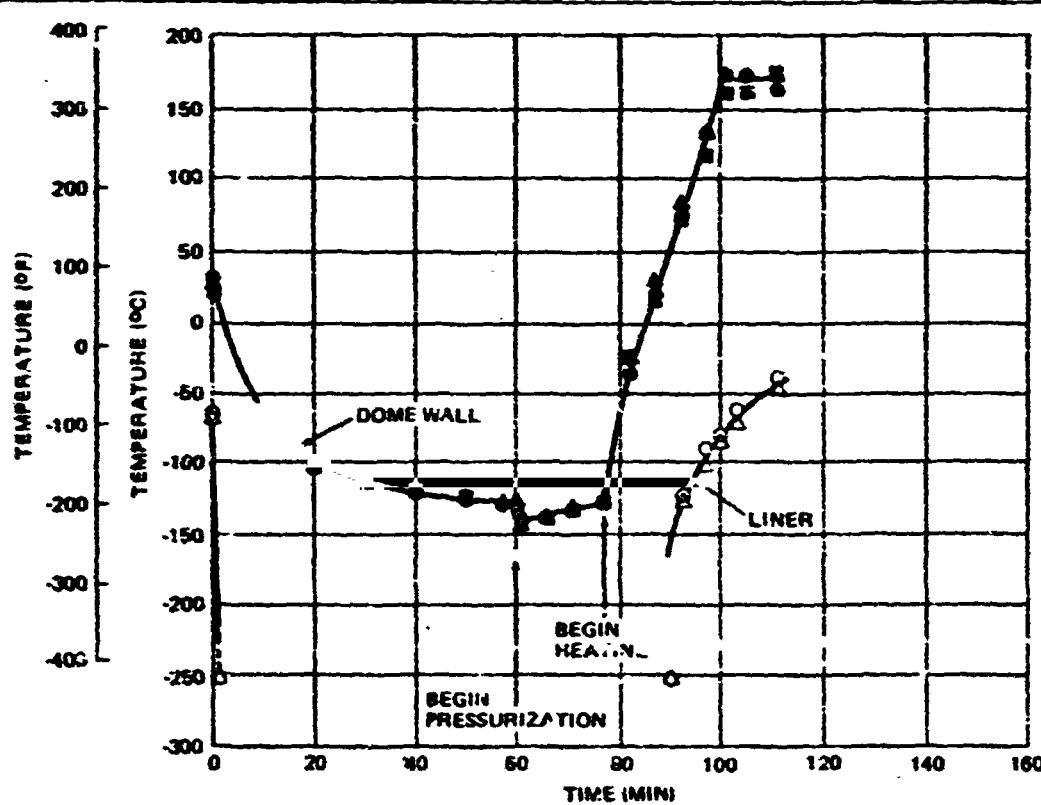


Figure 2-29. 1m (3 ft) Dome Test - Butt Joints - Cycle No. 6

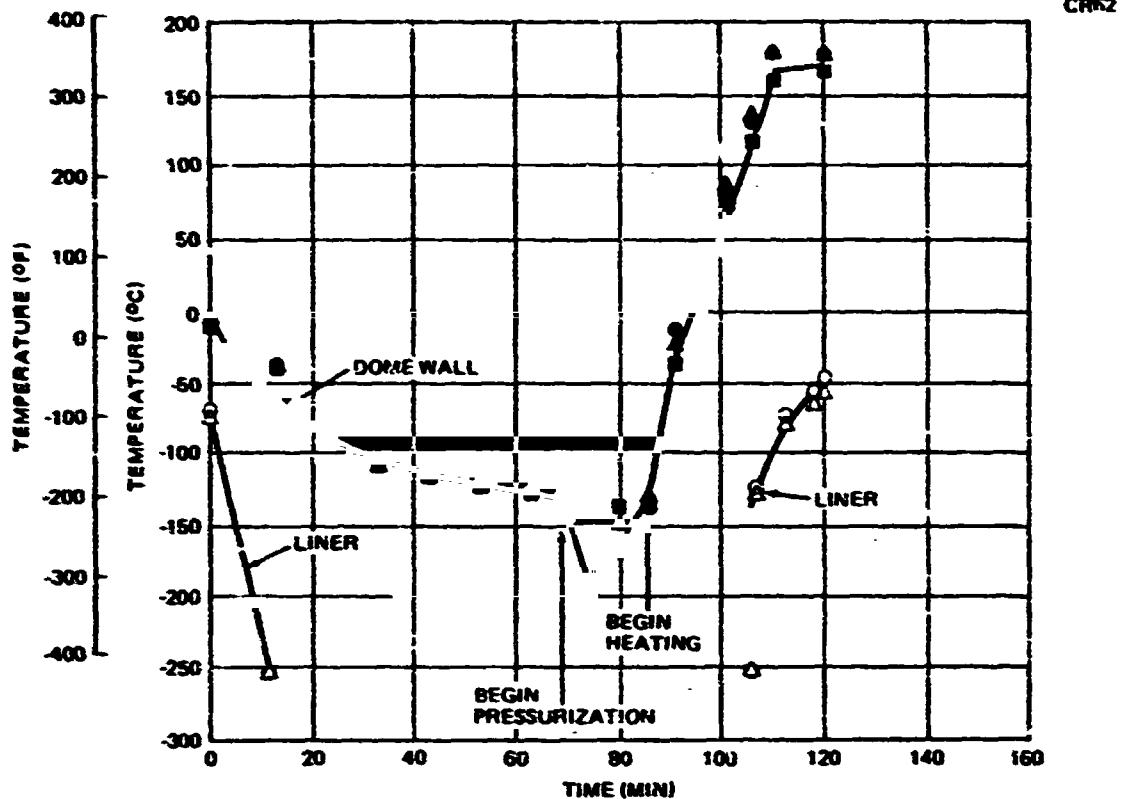


Figure 2-30. 1m (3 ft) Dome Test - Butt Joints - Cycle No. 7

### 2.5.3 Sonic Brush Examination

The equipment used for detecting debonds between the glass liner and 3D foam was an MDAC/NASA sonic delamination detector A-559-61819-1-PTE 901 shown in Figure 2-31. A reference panel of identical construction as each of the 1-m (3-ft) dome insulation configurations was fabricated and had debonded areas of 1.27-cm (0.5-in.) or 2.54-cm (1.0-in.) diameter. The battery-powered sonic brush was operated by pressing the wire brush lightly against the glass liner surface and moving laterally to develop a rasping sound that was amplified and received as audio response through an 8-ohm stereophonic headset receiver. An unbonded area produced a high-pitched sound level compared to the low rumble produced by a well-bonded area. The entire surface of the liner was examined before cycle testing. The usual debonded areas were found along the joint line between 3D foam pieces where the liner bridges the joint. These areas were less than

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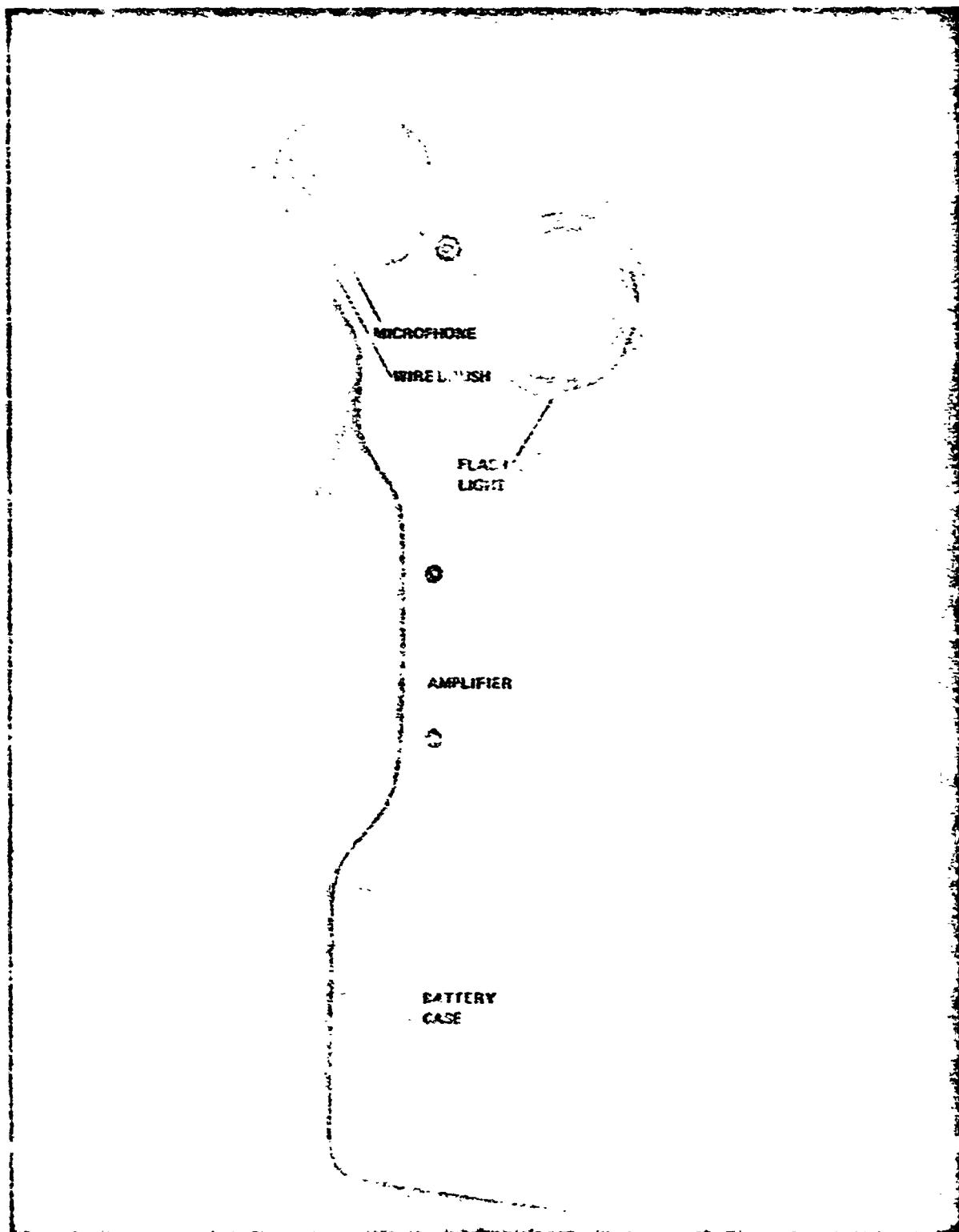


Figure 2-31. Sonic Delamination Detector A-659-61819-1-PTE901

0.64-cm (0.25-in.) wide by 2.54-cm (1-in.) long and were acceptable. No repair of the liner was needed before cycle testing.

After the first cycle, LH<sub>2</sub> under 0.21 MN/m<sup>2</sup>g (30 psig), the liner was examined visually and with the sonic brush. The liner was clearly strained by exposure to LH<sub>2</sub> under 0.21 MN/m<sup>2</sup>g, as evidenced by craze marks at the Z-thread contact points. The craze marks in the bonding resin fillets were visible only in the peripheral area of the dome within 10 cm (4 in.) of the attached flange.

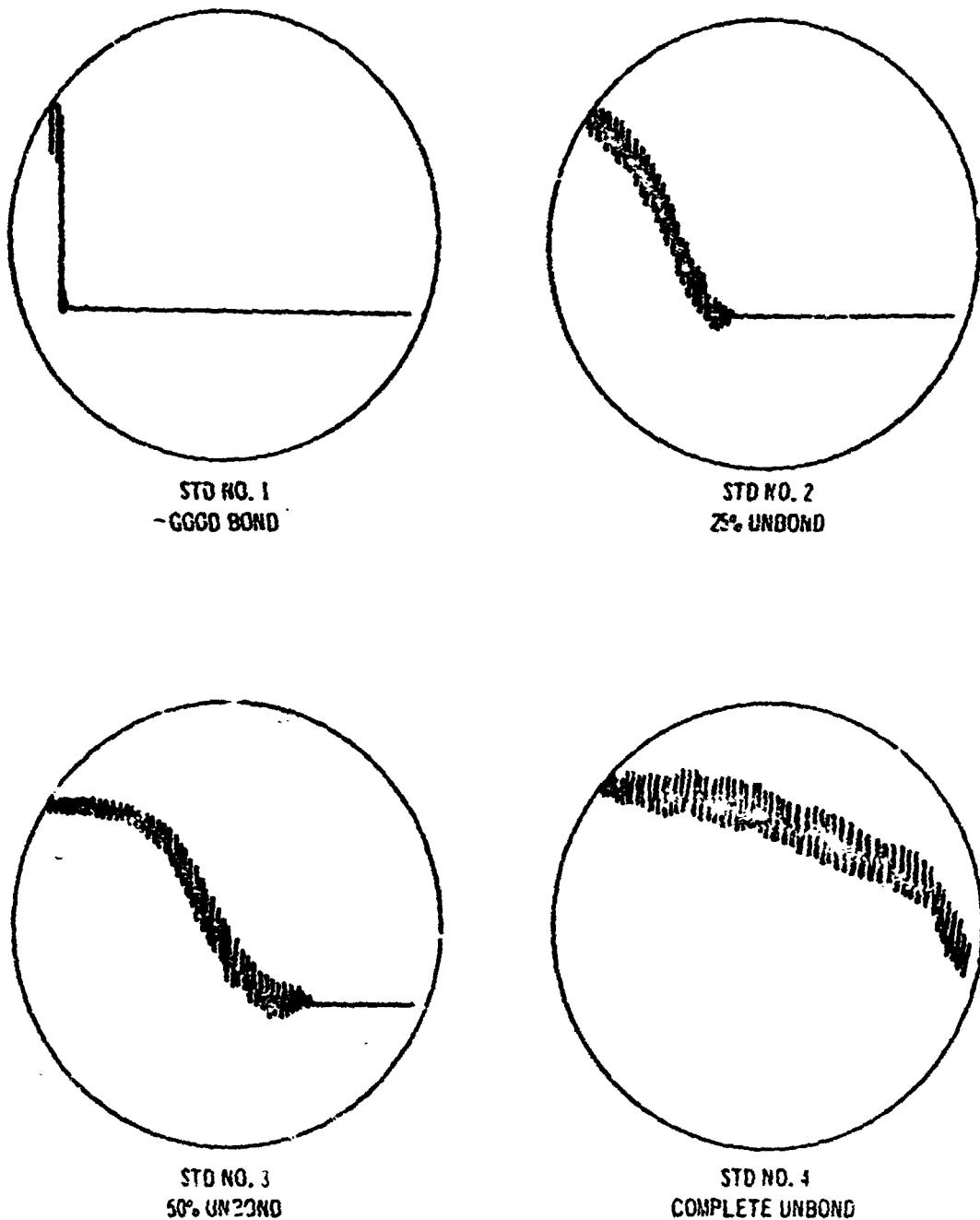
#### **2.5.4 Ultrasonic Examination**

The equipment used for pulse-echo ultrasonic examination of the L211A/LZ bond of the BX-251A-3D to the aluminum dome was:

- Reflectoscope UM-700-Sperry Products Company
- Transigate E550 Alarm-Sperry Products Company
- Transducers, 5.0 MC 1.9-cm (3/4-in.) Diameter LS Flat Contact - Sperry Products Company

A reference test panel of the same thickness, materials, and construction was fabricated, representing each dome insulation configuration having debonds of 1.27-cm (0.5-in.) and 2.54-cm (1-in.) diameter. The reference test panels also contained areas in which spots of adhesive, 0.64-cm (0.25-in.) square, were removed to provide "porous" areas of 25 and 50-percent debond. Figure 2-32 shows the reflectoscope patterns produced by complete debond as well as 25 and 50-percent porosity. A water/glycerine solution was brushed on the dome metal surface to provide a couplant between the transducer and dome surface.

The entire area of the dome was scanned prior to cycle testing and again after the 7 cycles. Only the areas under the ship lap joints in Dome No. 1 were found to be suspect and compared to the pattern indicating 25-percent porosity. No repair was indicated prior to cycle testing. After 7 cycles, these same areas were examined again and no growth could be found.



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Figure 2-32. Ultrasonic Patterns from Reference Standards Used for Evaluating Bond Condition

The ultrasonic registration observed while examining dome No. 2, low-weight adhesive, was not clearly defined between well-bonded adhesive areas and the areas having complete debond. In fact, the entire surface of dome No. 2, low-weight adhesive, registered as "porous" adhesive. The corresponding reference panel was used to reset the sensitivity of the ultrasonic unit to locate completely debonded areas. After the 7 test cycles in LH<sub>2</sub>, dome No. 2 still registered as having "porous" adhesive with no complete debond areas. As usual, the areas under the joints between 3D foam could be located by ultrasonic examination.

#### 2.5.5 Tensile Plug Testing of 3-Ft Dome

After conducting the two nondestructive examinations of the bonded insulation, a group of 3.8-cm (1.5-in.) diameter aluminum plugs were bonded to specific areas of the insulation using Lefkoweld 109/LM-52 adhesive cured at 25°C (77°F). The 3D foam-to-tank wall bond line tests were conducted by cutting and peeling off the liner to bond the aluminum plug directly to the 3D foam. The liner-to-3D foam bond tests were conducted by bonding the aluminum plugs directly on top of the glass liner.

The fixture for pulling the plugs and the instrument for load registration is described by Figure 2-33. Testing at -196°C (-320°F) on the liner was accomplished by flooding the dished liner surface with LN<sub>2</sub> for 5 minutes prior to pulling the plug. Testing at -196°C on the 3D foam-to-dome wall was accomplished by placing the dome, metal side down, in a flat tray filled with LN<sub>2</sub>. Figures 2-34 and 2-35 show the test setup for the plug tests. Table 2-5 presents the plug test data.

It should be noted that the small size of the plugs, 3.8-cm (1.5-in.) diameter [ $11.3\text{-cm}^2$  (1.77-in.<sup>2</sup>; cross-sectional area)] was selected to develop a minimum of 0.7-MN/m<sup>2</sup> (100-psi) tensile bond line stress without exceeding a load that would induce permanent deflection in the 0.11-cm (0.045-in.) thick aluminum dome skin. This size limitation placed the plug test under the disadvantage of having greater edge effect when compared to the MIL-STD-401 specimen tested in self-aligning load fixtures and having a 25.8-cm<sup>2</sup> (4-in.<sup>2</sup>) area. The MIL-STD-401 test method was used to determine tensile

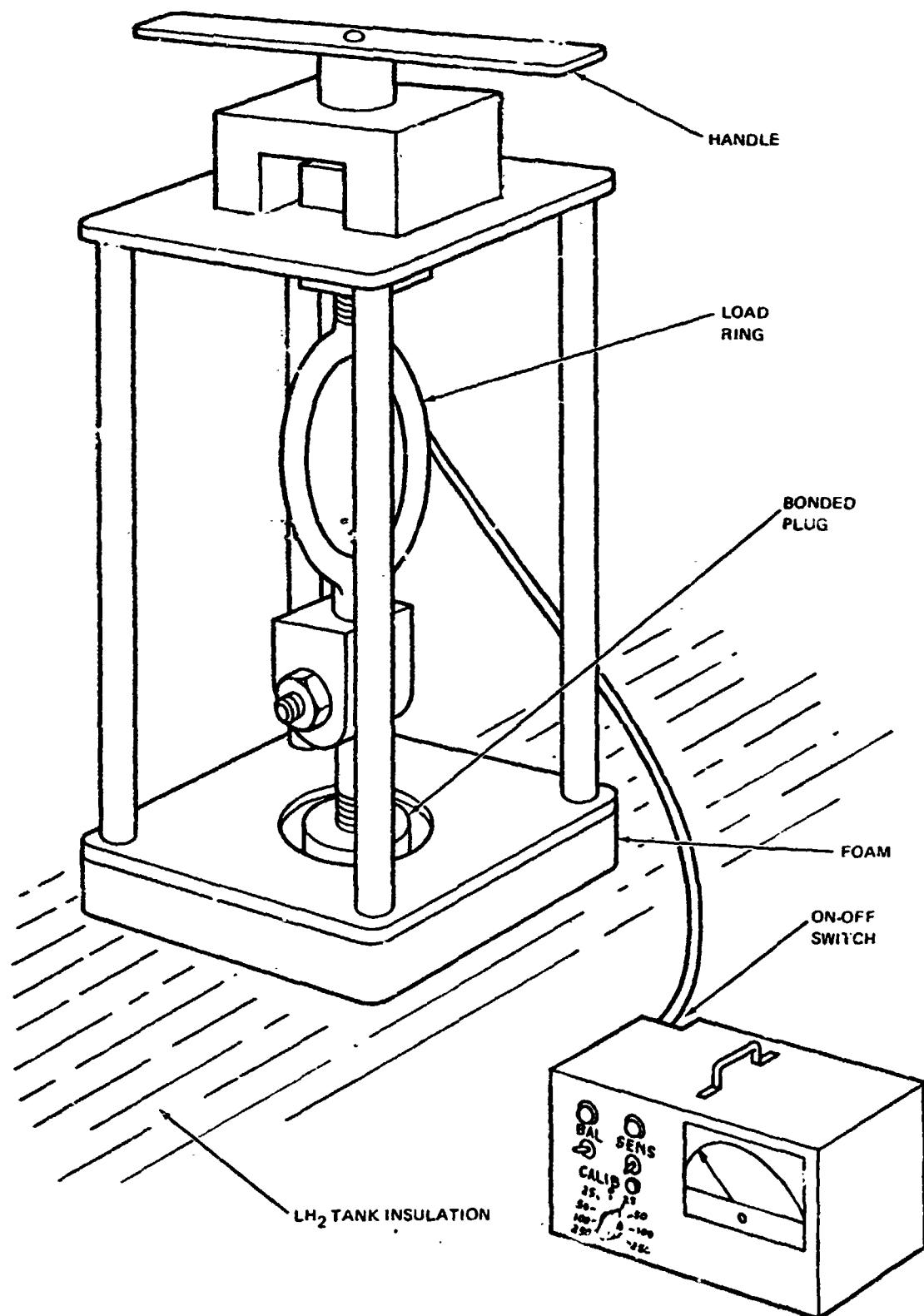


Figure 2-33. Test Hookup

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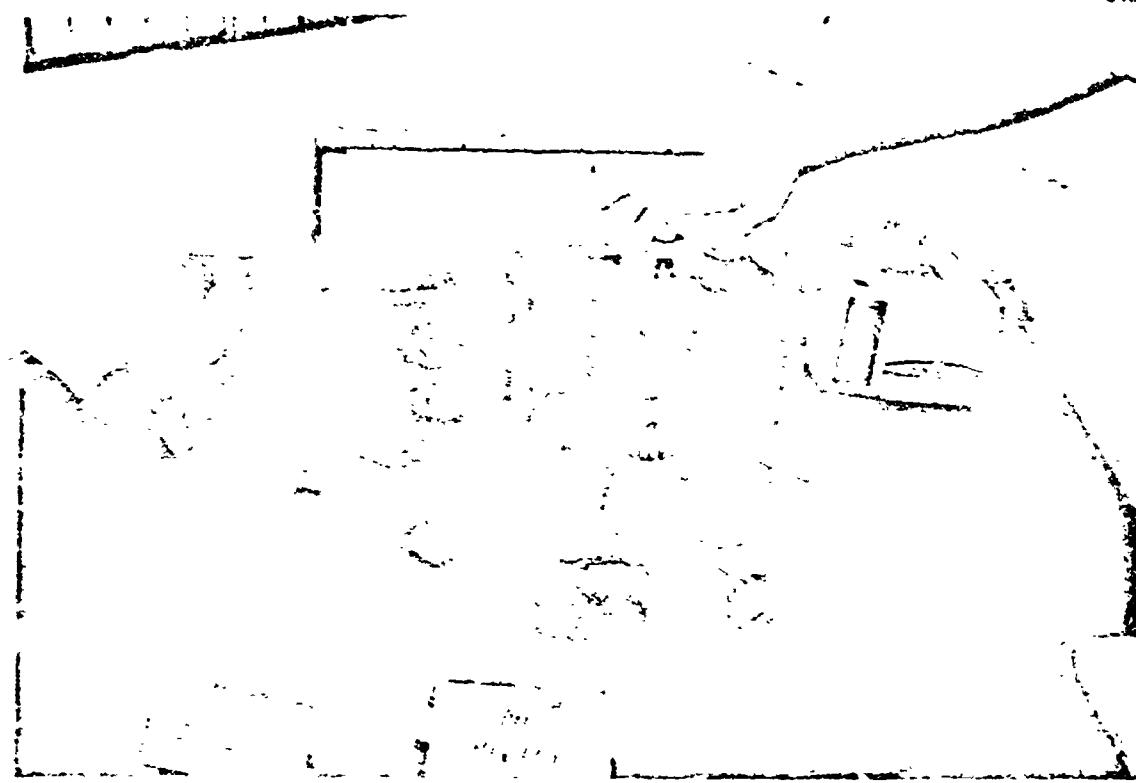


Figure 2-34. Tensile Plug Testing-Dome No. 1

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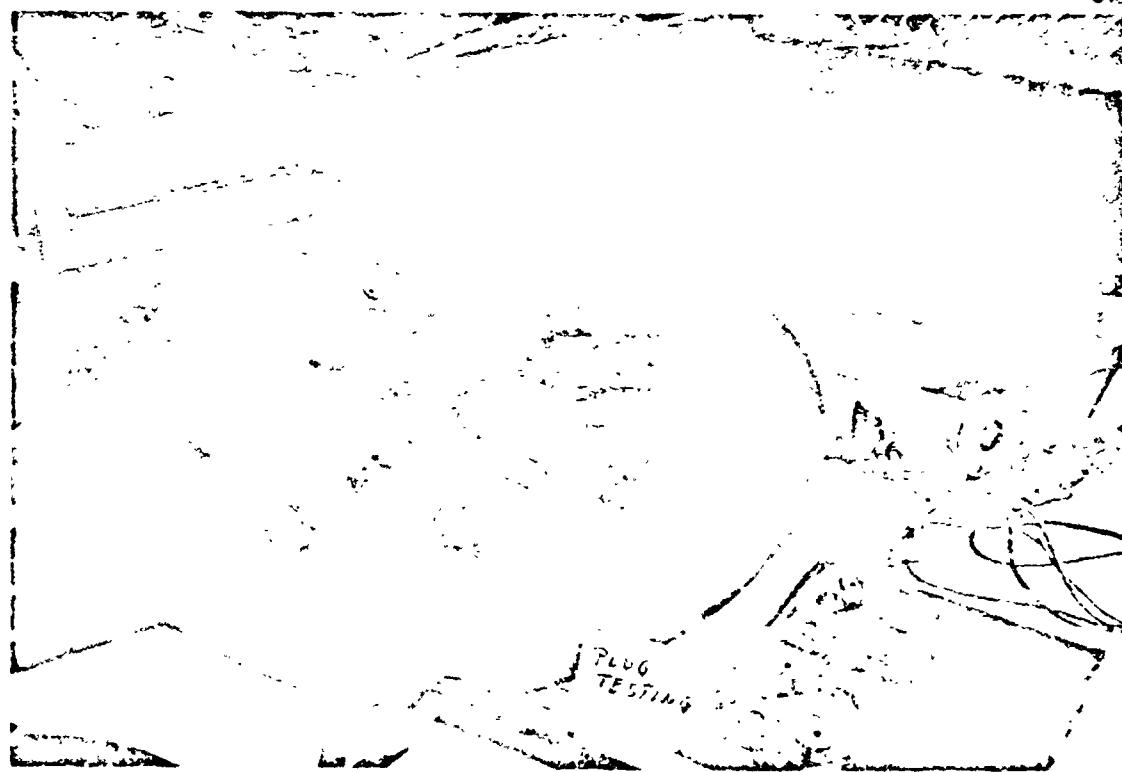


Figure 2-35. Tensile Plug Testing-Dome No. 2





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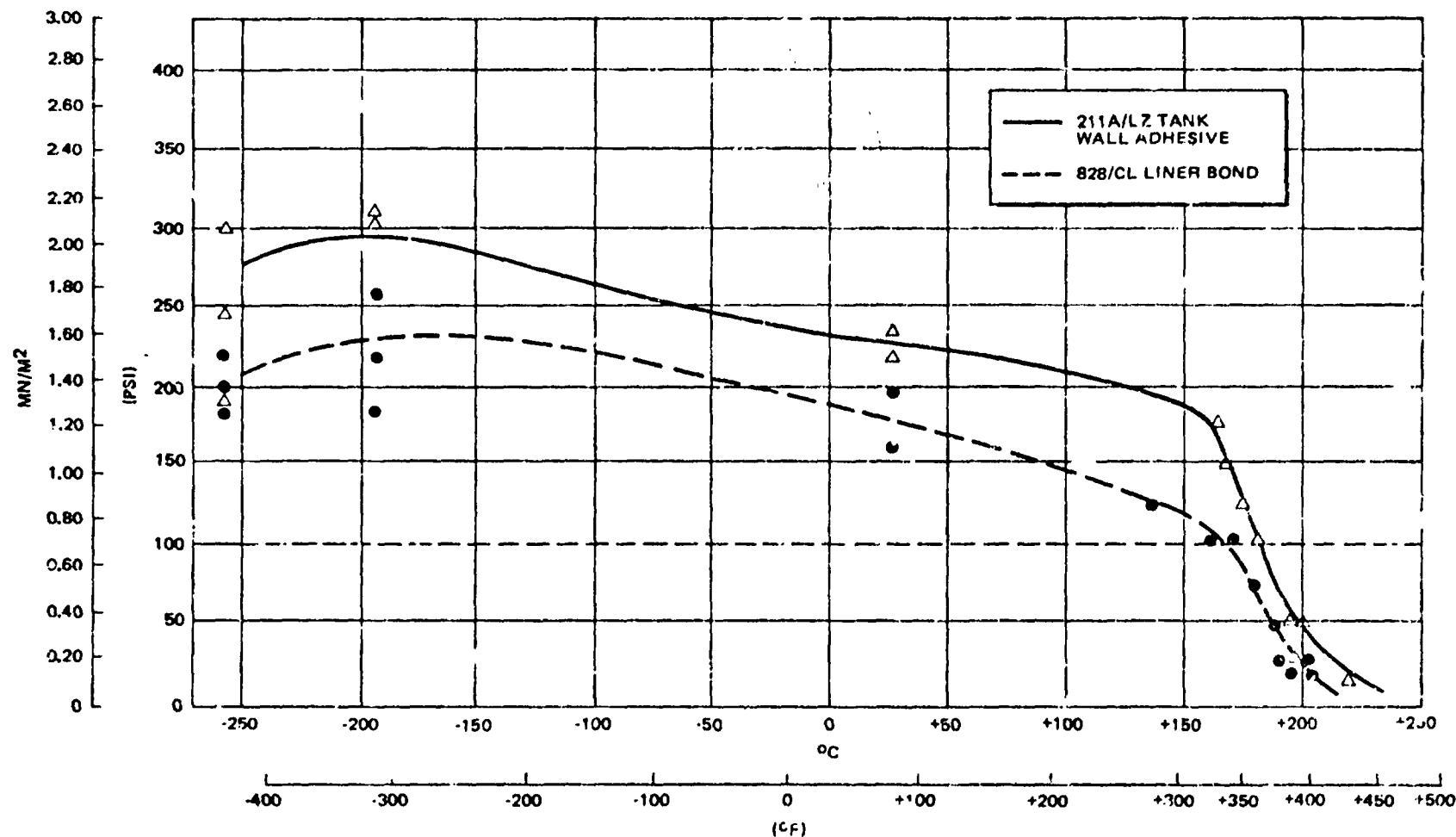


Figure 2-36. Terile Bond Strength -- Composite

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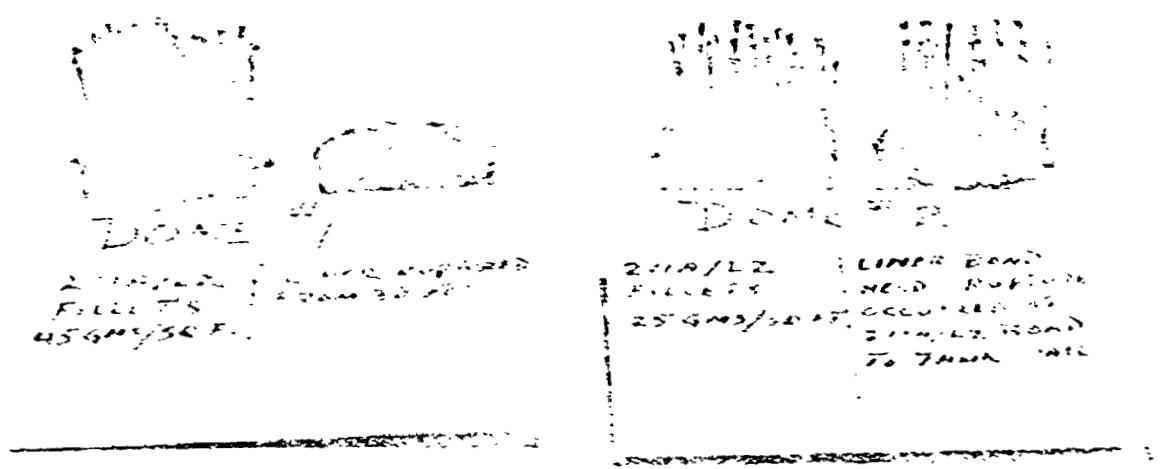


Figure 2-37. Adhesive Effects

reinforcement layer of 116 glass fabric but did not result in rupture of the glass fabric threads. These stress lines were not associated with butt joints in the 3D foam. In fact, two of the marks terminated at a butt joint, and obviously were not influenced by the low-weight adhesive bond between the 3D foam and the dome plate. For this reason, it is safe to assume the liner behaved in an acceptable manner and was not influenced by the syntactic foam used to bond the 3D foam joints or the low-weight adhesive against the 2219-T87 dome plate.

The liner-to-3D foam bond strength values were lower than might have been expected on both dome and flat panels but not abnormally so. This lead to the conclusion that the liner bond, using unfilled rigid epoxy resin, appeared to be more sensitive to the peel effects induced by plug testing than was the BX-251A-3D-to-tank wall bond.

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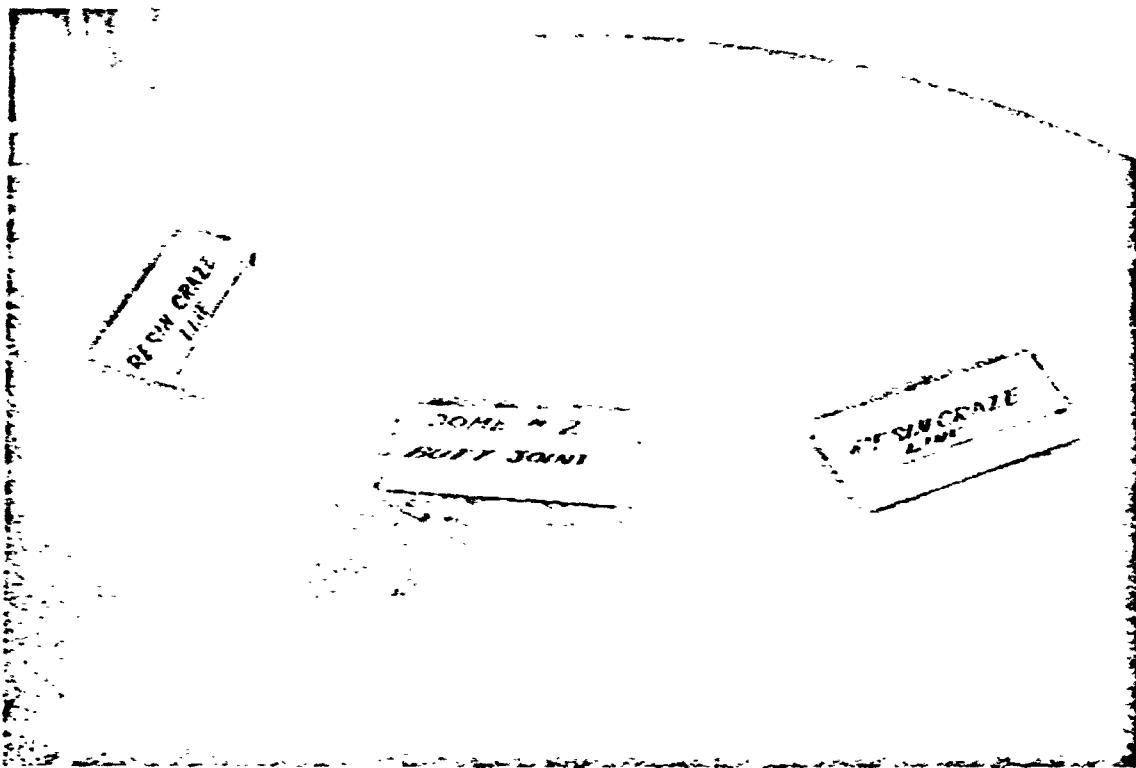


Figure 2-38. Dome No. 2 Resin Craze Mark

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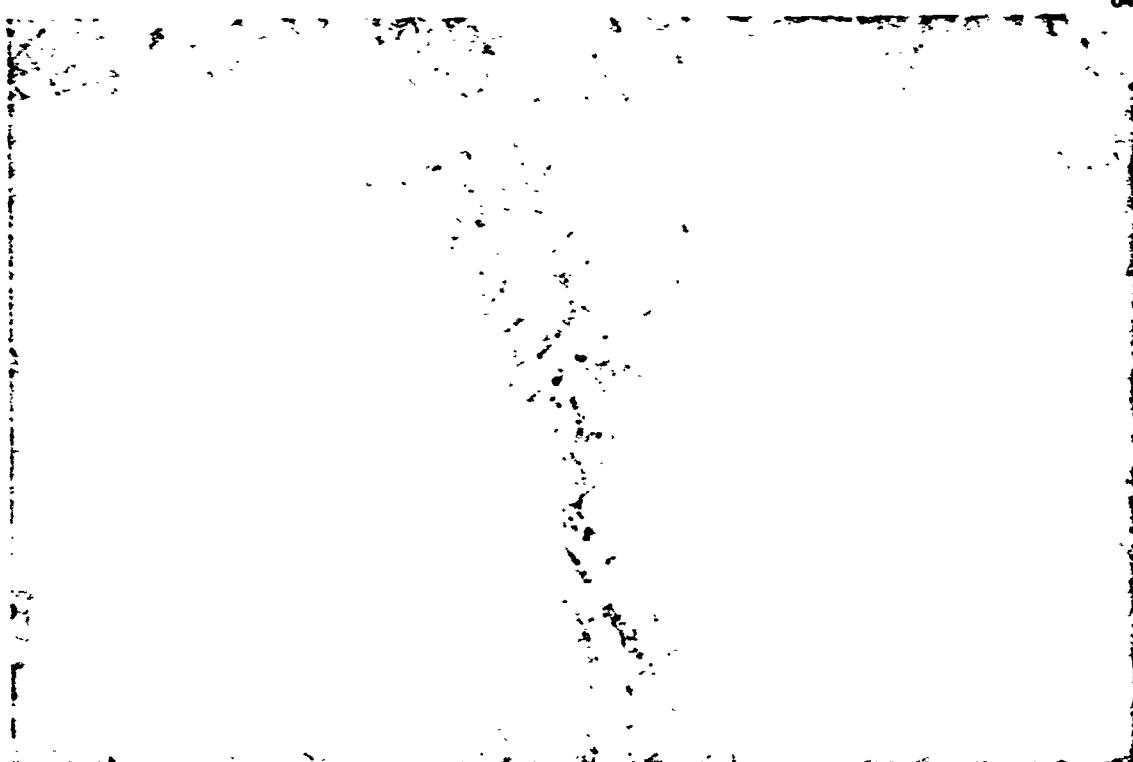


Figure 2-39. Closeup of Resin Craze Mark

In summation, results of plug tests conducted on the bond lines between glass liner-to-3D foam and the 3D foam-to-dome wall (Table 2-5) revealed no significant degradation effects that would indicate cause for corrective action, or that would detract from a high level of confidence in continuing cycle testing to 100 or more cycles.

The most significant observation associated with these dome tests is that dome No. 2, having low-weight adhesive and butt joints between 3D foam pieces, performed as efficiently as either of the preceding domes having twice the weight of adhesive at the metal bond line and the typical S-IVB style ship lap joints between 3D foam pieces. It is very interesting to note the best processing efforts to obtain large adhesive fillets on dome No. 2 using low-adhesive weight did not match the fillet size obtained with dome No. 1 using normal adhesive weight. This difference in fillet size is shown in Figure 2-37. Even so, the plug tests show the strength of the low-weight fillets to be in the same order of magnitude as that obtained with normal-weight fillets.

## 2.6 COST EFFECTIVENESS OF SHUTTLE 3D FOAM SYSTEM VERSUS SATURN S-IVB STYLE INSULATION

The S-IVB style insulation and the new Shuttle internal insulation consists of the following elements:

	S-IVB Style -423°F to +200°F	Shuttle Style -423°F to +350°F
Adhesive Tank wall-to-3D foam	Lefkoweld 109/LM-52 Epoxy-Aliphatic Amine	Lefkoweld 211A/LZ Epoxy-Aromatic Amine
Type 3D foam core	5.2 PCF density	2.7 PCF density
116 glass liner impregnation and bond-to-3D foam	Stabond U-135 or NARMCO 7343 with MOCA	EPON 828/CL Epoxy-Aromatic Amine

The key cost factor related to internal insulation bonding operations concerns the size of area or segment that can be covered with catalyzed adhesive-coated 3D foam and glass liner before vacuum-bag pressure must be applied. With the tank wall and environmental temperature of 285 to 290°K (55 to 65°F), the S-IVB insulation segments were seldom over 240 m<sup>2</sup> (22 ft<sup>2</sup>) in size and which were completed within a 5-hour bonding sequence. The cost (manhours) required to seal 27 individual vacuum bags inside the S-IVB tank comprised a major portion of the insulation cost.

During development of the Shuttle insulation system, the elevated temperature strength characteristics figured prominently in the selection of candidate adhesives to be tested; however, the working life of the catalyzed Lefkoweld 211A/LZ adhesive was found to be equally attractive. The catalyzed working life (in hours of layup time) of Lefkoweld 211A/LZ adhesive is over six times that of Lefkoweld 109/LM-52, allowing over 10 times more area to be insulated prior to applying vacuum-bag pressure. In addition, the glass liner, impregnated and bonded to the 3D foam using EPON 828/LL epoxy resin, possessed working-life characteristics very close to those of Lefkoweld 211A/LZ which allowed both bonding operations to be accomplished simultaneously under the same vacuum pressure bag.

Other features having an influence on the installation cost of applying internal insulation are noted in Table 2-6 in which a comparison between the S-IVB style and Shuttle insulations are presented. The tank size difference between the two vehicles has been considered in this comparison, but a larger tank area is of minor value in reducing cost if a short working life of the adhesives used prevents exploitation of the larger bonding area available.

The elapsed time for bonding insulation is also presented in Table 2-6. The elapsed times are listed as the lineal sequence time to serve as a direct comparison between the two insulation systems and does not include allowance for inspections, records, warming and cooling time, or any of the normally planned holding periods for instrumentation, installation, and insulation extensions. A significant reduction in elapsed time is possible using the Shuttle insulation system and this factor will allow more efficient use of the vehicles in assembly.

Table 2-6  
COST COMPARISON INTERNAL INSULATIONS

	S-IVB 3D Foam	Shuttle 3D Foam		
Installation cost	Tank size	Tank size		
Labor hours	6.7 m (22 ft) D x 9.5 m (31 ft) L (1 DOME)	7.6 m (25 ft) D x 24.4 m (80 ft) L (2 DOMES)		
• Slice 3D foam	Shiplap edges	Butt edges		
• Coat 3D foam with tank-wall adhesive	Machine mix and coat	Machine mix and coat		
• Impregnate glass fabric with resin	Hand mix and hand impregnate	Machine mix and roller impregnate		
• Position 3D foam and liner inside tank	Chain conveyor 2-man operation	Paper conveyor 1-man operation		
• Seal PVC film vacuum bay	27 separate bay operations 19.8 m <sup>2</sup> (220-ft <sup>2</sup> ) per bag	3 separate bay operations 243 m <sup>2</sup> (2,700 ft <sup>2</sup> ) per bag		
• Postcure cycle	24 hr at 71° C (160° F)	32 hr at 149° C (300° F)		
• Estimated operational hours				
Tank surface area insulated	17 man hr/m <sup>2</sup> (1.6 man hr per ft <sup>2</sup> )	Within 20% of actuals	5.1 man hr/m <sup>2</sup> (0.47 man hr per ft <sup>2</sup> )	No actuals available for comparison
• Estimated elapsed time for bonding insulation	22 days	Lineal sequence time only	7 days	Lineal sequence time only

### **Section 3**

### **SUMMARY AND CONCLUSIONS**

**The reporting period was marked by several significant accomplishments, observations, and conclusions.**

#### **System Evaluation Testing**

The new 3D foam internal insulation system was subjected to seven simulated (abort) mission cycles in 3-ft dome tests consisting of LH<sub>2</sub> tanking, pressurization, and reentry heating to +177°C (+350°F). Two different configurations were tested using separate dome plates. Dome No. 1 consisted of:

- BX-251A-3D foam having a density of 44 kg/m<sup>3</sup> (2.7 pcf) and configured with shiplap joints between pieces.
- Lefkoweld 211A/LZ tank wall adhesive having a density of 0.484 kg/m<sup>2</sup> (45 g/ft<sup>2</sup>) and deposited on the 3D form surface using a production-type automatic mixing and dispensing machine.
- A liner of 116 glass fabric impregnated and bonded to 3D foam using Epon 828/CL epoxy resin.

This insulation composite was bonded to the anodized 2219-T87 aluminum dome plate using the manufacturing methods (developed in Phase II of this project) that would be required for bonding large production-type LH<sub>2</sub> tanks. The in-process tests required for production operations were performed, and the limits of manufacturing tolerances were exercised. The successful completion of seven simulated mission cycles using Dome No. 1 (there were no evaluations) constituted the first step in validating the new materials and production methods for eventual use in the reusable LH<sub>2</sub> fuel tanks of the Space Shuttle.

Following this series of LH<sub>2</sub> cycle tests, another dome plate was insulated using a lower weight of adhesive per unit of surface area and a lower-cost configuration of 3D foam. Dome No. 2 consisted of:

- BX-251A-3D foam having a density of 44 kg/m<sup>3</sup> (2.7pcf) and configured with butt joints between pieces.
- A low-density joint filler having a density of 400 kg/m<sup>3</sup> (25 pcf) used between 3D foam pieces and at termination edges.
- Lefkoweld 211A/LZ adhesive, automatically machine-mixed but deposited primarily on the Z-thread ends and weighing only 0.27 kg/m<sup>2</sup> (25 g/ft<sup>2</sup>) (slightly over one-half the normal weight).
- A liner of 116 glass fabric impregnated and bonded to 3D foam using Epon 828/CL epoxy resin.

The same limits of adhesive working life, vacuum pressure, and cure schedule developed in Phase II of this project were exercised during bonding of the insulation in Dome No. 2. The lower-weight and lower-cost configuration of the insulation used in this dome also successfully completed seven simulated mission cycles—pressurization with LH<sub>2</sub> and heating to +177°C (+350°F)—with no significant thermal or structural degradation.

Although the dome test program was carried to only seven cycles on each of the domes, the severity of the simulated abort missions—in which the aluminum surface is heated while LH<sub>2</sub> is still covering the liner—lent confidence to the conclusion that this new insulation system would meet the 100-mission requirement without need for repair or refurbishment.

#### BX-251A-3D Foam

The successful completion of the LH<sub>2</sub> cycle tests on both dome plates permits a high level of confidence in the use of BX-251A foam as the supporting matrix for the 3D glass thread array. BX-251A is not only the most heat-resistant foam tested in this project; it also has the lowest density and a fine cell structure.

#### Low-Weight Gap Filler

Fortunately, the development and qualification of a low-weight gap filler also allows use of butt joints between 3D foam pieces. These joints will result in significant cost reduction for internal insulation of large LH<sub>2</sub> tanks.

#### Low Weight of Adhesive Per Unit of Surface Area

On a Shuttle LH<sub>2</sub> tank with a surface area of 929 m<sup>2</sup> (10,000 ft<sup>2</sup>), the adhesive deposited only on the Z-thread ends of the 3D foam will save over 500 pounds of weight. The method used to achieve adhesive fillets of acceptable size without leaving large amounts of adhesive on the foam between thread ends may be unique but can be adapted to production operations. The success of Dome No. 2 in LH<sub>2</sub> cycle testing substantiates the use of adhesive deposited only at the thread contact fillets, and by improving the method of deposition will allow further weight reduction without altering the structural or thermal efficiency of 3D foam used for internal insulation.

#### Achievement of Project Objectives

Successful systems testing has demonstrated that the original structural and thermal objectives of this project have been achieved. In the area of insulation weight and cost reduction, the original objectives have been exceeded.



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**Section 4  
REFERENCES**

- 1. O. Salmassy, et. al., Development of Advanced Materials Composites for Use as Insulators for LH<sub>2</sub> Tanks. MDAC Quarterly Summary Report MDC G2525, Contract NAS 8-25973, September 1971.**
- 2. McDonnell Douglas Astronautics Company. Space Shuttle Phase B Extension Study Plan, 19 July 1971.**
- 3. McDonnell Douglas Astronautics Company. External LH<sub>2</sub> Tank Study. Technical Summary Report EO 376, 30 June 1971.**
- 4. C. R. Lemons, C. R. Watts, and O.K. Salmassy. Development of Advanced Materials Composites for Use as Insulators for LH<sub>2</sub> Tanks. Summary Report Phase II MDC G3677, Contract NAS 8-25973, June 1972.**



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**Appendix**  
**MATERIAL AND PROCESS SPECIFICATIONS**

## FOAM, POLYURETHANE, CRYOGENIC

### 1. SCOPE

1. 1 Scope—This drawing covers the minimum requirements for one type of cryogenic polyurethane foam used in structural support and thermal insulation in applications where temperatures are in the range of -253C (-423F) to 177C (350F).

### 2. APPLICABLE DOCUMENTS

2. 1 The following specifications and standards (and subsidiaries thereof), drawings and publications of issue in effect on date of invitation for bid, except as otherwise noted or controlled on an individual basis, form a part of this drawing to the extent specified herein.

### SPECIFICATIONS

#### FEDERAL

PPP-B-576	Box, Wood, Cleated, Veneer, Paper Overlaid
PPP-B-591	Boxes, Fiberboard, Wood-Cleated
PPP-B-601	Boxes, Wood, Cleated - Plywood
PPP-B-621	Boxes, Wood, Nailed and Lock-Corner
PPP-B-636	Box, Fiberboard
PPP-B-640	Boxes, Corrugated, Triple Wall, 350 Pound Maximum Weight
PPP-C-96	Cans, Metal, 28 Gage and Lighter
PPP-P-704	Pails, Shipping, Steel, (1 through 12 gallon)

### STANDARDS

#### MILITARY

MIL-STD-129	Marking for Shipment and Storage
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The following statement is applicable only for direct Government contracts: Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting activity.

MCDONNELL DOUGLAS ASTRONAUTICS CO.  
HUNTINGTON BEACH, CALIF.

MCDONNELL DOUGLAS

50-0213-1A02 '96 AUG '71

SIZE <b>A</b>	CODE IDENT NO. <b>18355</b>	DRAWING NO.
SCALE	1/16"	SHEET

**2.2 Other publications**—The following publications form a part of this drawing. Unless otherwise indicated, the issue in effect on date of invitation for bid shall apply.

**AMERICAN SOCIETY FOR TESTING AND MATERIALS**

**ASTM C177**

**Method of Test for Thermal Conductivity of Materials by Means of the Guarded Hot Plate**

**ASTM D1621**

**Compressive Strength of Rigid Cellular Plastics**

**ASTM D1622**

**Apparent Density of Rigid Cellular Plastics**

**ASTM D1623**

**Tensile Properties of Rigid Cellular Plastics**

**3. REQUIREMENTS**

**3.1 Preproduction approval**—When preproduction approval is required by the contract or order the material furnished under this drawing for preproduction approval shall be a product which has been tested and has passed the preproduction tests specified herein. After preproduction approval, the properties and method of manufacture shall not be changed without written approval from the procuring activity.

**3.1.1 Preproduction sample**—When preproduction approval is required, samples submitted shall be for preproduction tests outlined in this drawing. Unless otherwise specified, sample shall consist of one liter (quart) of the isocyanate with the correct proportion of polyol to mix per manufacturer's recommendations. Samples shall be marked with the material requirements drawing title, number, change letter, isocyanate or polyol, manufacturer's name and designation, date of manufacture, toxicity warning and instructions for mixing.

**3.2 Material**—The material used in the manufacture of the product shall be of uniform quality, suitable for the intended purpose. The material shall be supplied as a two component liquid system and when mixed in the proper proportions and cured shall produce a methyl glucoside copolymerized polyurethane foam. A halocarbon (Freon 11, Ucon 11, or equal) shall be the blowing agent.

**3.3 Storage life**—Unless otherwise specified, the storage life of the unmixed components shall be a minimum of 12 months from date of manufacture when stored in a closed container at a temperature of 21C & 70F to 27C (80F) and no material shall be supplied with less than 11 months shelf life.

**3.4 Cure**—The material shall have a handling cure, when mixed per manufacturer's instructions, in a maximum of 15 minutes and at a maximum temperature of 27C ± 2C (80F ± 3F).

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MCDONNELL DOUGLAS

SIZE	CODE IDENT NO.	DRAWING NO.
A	18355	
SCALE	REV	SHEET

3.5 Density—The density of cured foam shall be  $24 \pm 3 \text{ kg/m}^3$  ( $1.5 \pm 0.2 \text{pcf}$ ).

3.6 Compression flatwise—The cured foam shall have a minimum compressive strength at 10 percent deflection of  $0.14 \text{ MN/m}^2$  (20 psi) when the loading is parallel to the direction of foam rise.

3.7 Tension flatwise—The cured foam shall have an average tensile strength of  $0.21 \text{ MN/m}^2$  (30 psi) when the loading is parallel to the direction of foam rise.

3.8 Thermal conductivity—The cured foam, shall have a maximum thermal conductivity of  $0.0005 \text{ cal/sec-cm-}^\circ\text{C}$  ( $0.25 \text{ BTU-IN/HR-FT}^2-{}^\circ\text{F}$ )

3.9 Heat stability—The foam shall have less than 12 percent weight loss when heated 100 hours at  $177^\circ\text{C}$  ( $350^\circ\text{F}$ ).

3.10 Product marking—The containers for material shall be durably and legibly marked with the material requirements drawing number, title, change letter, manufacturer's name and designation, batch number, date of manufacture, toxicity warning, isocyanate of polyol as applicable with mixing percentage and "STORE AT  $21^\circ\text{C}$  to  $27^\circ\text{C}$  ( $70^\circ\text{F}$  to  $80^\circ\text{F}$ )."

3.11 Workmanship—The materials shall be mixed and processed in accordance with the best practice for the manufacture of a high quality polyurethane foam. The material shall be free of foreign matter, sedimentation, lumps, and crystallinity.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the procuring activity. Inspection records of the examination and tests shall be kept complete and available to the procuring activity as specified in the contract or order. The procuring activity reserves the right to perform any of the inspections set forth in the drawing where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot—For purposes of sampling, inspection, and tests, a lot shall consist of the material manufactured at one time in one batch and offered for acceptance.

4.3 Sampling—Samples representing each lot of material in a shipment shall be used for inspection and testing to the requirements of this drawing.

#### 4.4 Classification of tests -

4.4.1 Preproduction—Preproduction tests enable the procuring activity to determine that the material complies with the drawing requirements.

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SCALE	REV	SHEET		
60-0213-1A02 (D8-AUG-71)				

4.4.1.1 Tests—The preproduction tests shall consist of all the tests described in 4.5 and 4.6 of this drawing.

4.4.2 Production—Production tests are those tests which shall be conducted by the manufacturer to assure conformity to the requirements of this drawing and enable the manufacturer to certify the accuracy of his product to the procuring activity.

4.4.2.1 Tests—The following tests shall be recorded in accordance with 4.1.

Paragraph

(a) Density	4.5.1
(b) Compression Flatwise	4.5.3
(c) Tension flatwise	4.5.4
(d) Examination	4.6

4.5 Test methods and procedures—

4.5.1 Density—Pour the calculated weight of foam mixed according to the manufacturer's instructions into an open 30.5 cm (12 in.) by 30.5 cm mold. After curing per 3.4, remove the block from mold. Allow to cool a minimum of 8 hours, cut off the top crown and approximately 1.3 cm (0.5 in.) layers from the sides and bottom, and discard the scrap pieces. The density of the foam shall be measured per ASTM D1622.

4.5.2 Preparation of test specimens—Specimens shall be prepared from the block prepared in 4.5.1. All specimens for test shall be free of skin. Unless otherwise specified, a minimum of three specimens shall be tested per each test specified herein.

4.5.3 Compression flatwise test—Specimens shall be fabricated in such a manner that the direction of loading is parallel to the direction of foam rise. The compressive strength of the foamed material shall be measured per ASTM D1621, procedure "A" to determine conformance to 3.6.

4.5.4 Tensile strength determination—Specimens shall be fabricated in such a manner that the direction of loading is parallel to the direction of foam rise. The tensile strength of the foamed material shall be measured per ASTM D-1623 to determine conformance to 3.7.

4.5.5 Thermal conductivity—Two specimens of cured foam shall be tested as specified in ASTM C177 to determine conformance with 3.8.

4.5.6 Heat stability test—Weighed specimens shall be heated in a convection type oven at  $177 \pm 2^\circ\text{C}$  ( $350 \pm 5^\circ\text{F}$ ) for 100 hours. The weight loss shall be measured and calculated as percent of original weight to determine conformance to 3.9.

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SCALE	REV	SHEET		
6J-0213-1A02 (06 AUG 71)				

**4.6 Examination -**

**4.6.1 Inspection of product -** The material shall be examined as necessary to determine conformance with the applicable requirements of 3.10 and 3.11.

**4.6.2 Inspection of packaging -** The supplier shall make such inspections as are necessary to assure that the requirement for preservation, packaging, packing and marking are met.

**5. PREPARATION FOR DELIVERY**

**5.1 Preservation and packaging -**

**5.1.1 Level A -**

**5.1.1.1 Isocyanate component -** The isocyanate component shall be packaged in 1-gallon or smaller size cans conforming to PPP-C-96 Type V, Class 2, or 5-gallon containers to PPP-P-704 Type II, Class 1. The container size shall be as specified on the contract or order.

**5.1.1.2 Polyol component -** The polyol component shall be packaged in cans of proportionate size to that of the isocyanate. One-gallon or smaller size cans shall conform to PPP-C-96 Type V, Class 4 and over one-gallon shall conform to PPP-P-704 Type I, Class 1.

**5.1.2 Level C -** Materials shall be packaged in accordance with the supplier's commercial practice.

**5.1.3 Unless otherwise specified in the contract or order, Level C preservation and packaging shall be provided**

**5.2 Packing -**

**5.2.1 Level A -** The liquid components, packaged as specified, shall be packed in containers conforming to any one of the following box specifications:

<u>Specification</u>	<u>Type or Class</u>
PPP-B-576	Class 2
PPP-B-591	Overseas Type
PPP-B-601	Overseas Type
PPP-B-621	Class 2
PPP-B-636	Class 2
PPP-B-640	Class 2

Sealing, closure, strapping and weight shall conform to the appendix of the applicable box specification.

MCDONNELL DOUGLAS ASTRONAUTICS CO. McDONNELL DOUGLAS	SL A	CODE IDENT NO. 13355	DRAWING NO.
	SCALE	REV	SHEET
65-0217 FAC. 10, AUG 71.	43-06		

5.2.2 Level B—The liquid components, packaged as specified, shall be packed in accordance with the domestic requirements of the appendix of PPP-C-96, or PPP-P-704, as applicable.

5.2.3. Level C—The liquid components, packaged as specified, shall be packed in a manner to assure carrier acceptance and safe delivery at destination. The containers shall be in accordance with the rules and regulations of the carrier applicable to the mode of transportation.

5.2.4 Unless otherwise specified in the contract or order, Level C packing shall be provided.

5.3 Marking—In addition to any special marking required by the contract or order, unit packages, intermediate packages, shipping containers for shipment to the Government, shall be marked in accordance with the requirements of MIL-STD-129.

5.3.1 Special marking—The following marking shall be applied to the interior and exterior containers:

**CAUTION:**

STORE AT 21C to 27C (70F to 80F)

**TOXIC:**

PROVIDE ADEQUATE VENTILATION DURING APPLICATION  
AVOID PROLONGED INHALATION OF VAPOR

The characters shall be a minimum of 1 cm (3/8 in.).

6. NOTES

6.1 Intended use—This material is intended primarily for neutral or reducing cryogenic fluid applications on space vehicles.

6.2 Ordering data—Orders or contracts should specify the following information:

- Title, number and change letter of this drawing
- Level of packaging and packing required and container size.
- Options offered, if required (see 3.1.1, 3.3, 4.1, 5.1.3 and 5.2.4).

6.3 Caution—This material is not to be used in storage of oxidizing fluids.  
Use only for storage of neutral or reducing cryogenic fluids.

MCDONNELL DOUGLAS ASTRONAUTICS CO. MONTGOMERY BEACH, CALIF. <i>[Signature]</i>	SIZE	CODE IDENT NO.	DRAWING NO.
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	SCALE	REV	SHEET

## FOAM, POLYURETHANE, YARN REINFORCED

### 1. SCOPE

1.1 Scope — This drawing covers the minimum requirements for a yarn-reinforced polyurethane foam used as structural support and thermal insulation in applications where operating temperatures are in the range of -253C (-423F) to 177C (350F).

### 2. APPLICABLE DOCUMENTS

2.1 The following specifications and standards (and subsidiaries thereof), drawings and publications of issue in effect on date of invitation for bid, except as otherwise noted or controlled on an individual basis, form a part of this drawing to the extent specified herein.

### SPECIFICATIONS

#### FEDERAL

PPP-B-576	Box, Wood, Cleated, Veneer, Paper Overlaid
PPP-B-591	Boxes, Fiberboard, Wood-Cleated
PPP-B-601	Boxes, Wood, Cleated - Plywood
PPP-B-621	Boxes, Wood, Nailed and Lock-Corner
PPP-B-636	Box, Fiberboard
PPP-B-640	Boxes, Corrugated, Triple Wall, 350 Pound Maximum Weight

### STANDARDS

MIL-STD-129	Marking for Shipment and Storage
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The following statement is applicable only for direct Government contracts: Copies of specifications, standards, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting activity.

2.2 Other publications — The following publications form a part of this drawing. Unless otherwise indicated, the issue in effect on date of invitation for bid shall apply.

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60 0213-1A02 (06 AUG 71)				

## DRAWINGS

IP20011

## **Foam, Polyurethane, Cryogenic**

IP20019

## **Glass Fiber Yarn, Continuous, Structural**

(Application for copies of the above drawings should be made to the  
Douglas Aircraft Company, Inc., 3900 Ocean Park Boulevard,  
Santa Monica, California 90406.)

**AMERICAN SOCIETY FOR TESTING AND MATERIALS**

ASTM D1621

## **Compressive Strength of Rigid Cellular Plastics**

ASTM D1622

## Apparent Density of Rigid Cellular Plastics

(Application for copies of ASTM Standards should be addressed to the American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania 19103.)

### 3. REQUIREMENTS

3.1 Preproduction approval - The material furnished under this drawing for pre-production approval shall be a product which has been tested and has passed the preproduction tests specified herein. After preproduction approval, the properties and method of manufacture shall not be changed without written approval from the procuring activity.

**3.1.1 Preproduction sample** - A sample shall consist of a 15.3 x 30.6 x 30.6 cm (6 x 12 x 12 in.) block of the product. It shall be marked with the material requirements drawing title, number, change letter, and manufacturer's name and designation.

**3.2 Material** - The material used in the manufacture of the product shall consist of rigid polyurethane foam reinforced with a three dimensional mat of glass fiber yarn in the three axes.

3.2.1 The yarn in the "X" and "Y" axes shall conform to IP20019, Type II, Class 2.

3.2.2 The yarn in the "Z" axis shall conform to IP20019, Type III.

3.2.3 The polyurethane foam shall conform to (TSD), and the direction of foam rise shall be parallel to the "Z" axis.

**3.3 Density** – The density of the product shall be  $43 \pm 5 \text{ kg/m}^3$  ( $2.7 \pm 0.3 \text{pcf}$ ).

**3.4 Porosity** – The foam shall be predominately closed cell and a 2.54 cm (1 in.) thick specimen shall develop a minimum pressure drop of  $0.011 \text{ MN/m}^2$  (1.5 psi).

3.5 Compressive strength - The material shall have a minimum compressive strength at 10 percent deflection of 0.45 MN/m<sup>2</sup> (65 psi) at 25C (77F) when the loading is parallel to the direction of foam rise.

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**3.6 Heat stability** — The material shall have less than 16 percent weight loss when heated 100 hours at 177°C (350°F).

**3.7 Yarn spacing —**

**3.7.1** The yarn in the "X", "Y" and "Z" axes shall be located on nominal 0.46 cm (0.18 in.) centers.

**3.7.2** There shall be a minimum of 4 yarn ends per square cm (26 yarn ends per square inch) uniformly distributed in the "Z" axis. Parts which have ship-lap or tapered edges 0.64 cm (0.25 in.) thick or less may have a minimum of 3 yarn ends per square cm (18 yarn ends per square inch) in these edges.

**3.8 Fabricated parts** — After machining, at least 90 percent of the yarn ends in the "Z" axis shall protrude above the surface of the foam  $0.08 \pm 0.04$  cm ( $1/32 \pm 1/64$  in.).

**3.9 Blow holes** — Blow holes shall not exceed 0.24 cm (0.094 in.) in diameter, and there shall be not more than six such holes in an area of 6.45 square cm (ten square inches.).

**3.10 Workmanship** — The materials shall be processed in the best practice for the manufacture of a high quality polyurethane foam. There shall be no accumulations of unexpanded resin, tackiness, or charring, and the material shall be visually free of any foreign matter.

**4. QUALITY ASSURANCE PROVISIONS**

**4.1** The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the procuring activity. Inspection records of the examination and tests shall be kept complete and available to the procuring activity as specified in the contract or order. The procuring activity reserves the right to perform any of the inspections set forth in the drawing where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

**4.2 Lot** — For purposes of sampling, inspections, and tests, a lot shall consist of all the material manufactured and submitted for production at one time.

**4.3 Sampling** — Samples representing each lot of material shall be used for inspection and testing to the requirements of this drawing.

**4.4 Classification of tests —**

**4.4.1 Preproduction** — Preproduction tests enable the procuring activity to determine that the material complies with the drawing requirements.

**4.4.1.1 Preproduction tests** — The preproduction tests shall consist of all the tests described in **5.3** and **5.4.c** of this drawing.

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4.4.2 Production - Production tests are those tests which shall be conducted by the manufacturer to assure conformity to the requirements of this drawing and enable the manufacturer to certify the accuracy of his product in the procuring activity.

4.4.2.1 Production tests - The following tests shall be made as production tests.

<u>Test</u>	<u>Paragraph</u>
(a) Density	4.5.2
(b) Porosity	4.5.3
(c) Examination	4.6

4.5 Test methods and procedures -

4.5.1 Preparation of test specimens - Specimens shall be prepared from the product, and shall be free of all skin and molded surfaces. The thickness of all specimens shall be parallel to the direction of foam rise.

4.5.2 Density - The density shall be determined on a specimen measuring approximately  $15.3 \times 30.6 \times 30.6$  cm ( $6 \times 12 \times 12$  in.). Calculate the density per ASTM D1622 to determine conformance to 3.3.

4.5.3 Porosity test - The specimens shall be  $2.54 \pm 0.33$  cm ( $1 \pm 0.13$  in.) thick by a minimum of 20 cm (8 in.) square.

4.5.3.1 Install a 1.3 cm (0.5 in.) I.D. rubber base, approximately 3 m (10 ft.) long between a 15 CFM capacity vacuum pump and the 1.3 cm diameter end of the specimen holder fitted with a vacuum gauge. The opposite opening shall be  $17.7 \pm 0.3$  cm ( $7 \pm 0.125$  in.) diameter. Seal that 1.3 cm thick specimen over the 17.8 cm diameter opening. At full vacuum pump capacity the vacuum gauge attached to the specimen holder shall be read to determine conformance to 3.4.

4.5.4 Compressive strength test - The specimens shall be  $2.54 \pm 0.08$  cm ( $1 \pm 0.03$  in.) thick by  $5.08 \pm 0.08$  cm ( $2 \pm 0.3$  in.) by  $5.08 \pm 0.08$  cm ( $2 \pm 0.3$  in.) with the thickness parallel to direction of foam rise. A minimum of 5 specimens shall be tested at  $25 \pm 2$ C ( $77 \pm 5$ F) per ASTM D1621, procedure "A" to determine conformance to 3.5.

4.5.5 Heat stability test - Weighed specimens shall be heated in a convection type oven at  $177^{\circ}\text{C} \pm 2^{\circ}\text{C}$  ( $350^{\circ}\text{F} \pm 5^{\circ}\text{F}$ ) for 100 hours. The weight loss shall be measured and calculated as percent of original weight to determine conformance to 3.6.

4.6 Examination -

4.6.1 Inspection of product - Each slice and all test specimens of thread-reinforced foam shall be examined to determine conformance with the applicable requirements of 3.2, 3.7, 3.8, 3.9 and 3.10.

4.6.2 Inspection of packaging - The supplier shall make such inspections as may be necessary to assure that the requirements for preservation, packaging, packing and marking are met.

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## **5. PREPARATION FOR DELIVERY**

### **5. 1 Preservation and packaging -**

**5. 1. 1 Level A** - Material shall be packaged in accordance with the supplier's best commercial practice.

### **5. 2 Packing -**

**5. 2. 1 Level A** - Material, packaged as specified, shall be packed in containers conforming to any of the following box specifications:

<b>SPECIFICATION</b>	<b>TYPE OR CLASS</b>
PPP-B-576	Class 2
PPP-B-591	Overseas Type
PPP-B-601	Overseas Type
PPP-B-621	Class 2
PPP-B-636	Class 2
PPP-B-640	Class 2

Sealing, closure, strapping and weights shall conform to the appendix of the applicable box specification.

**5. 2. 2 Level B** - Material, packaged as specified, shall be packed in containers conforming to any of the box specifications listed in 5. 2. 1. Sealing, closure, strapping, and weights shall conform to the appendix of the applicable box specification.

**5. 2. 3 Level C** - Material, packaged as specified, shall be packed in a manner to insure carrier acceptance and safe delivery at destination. The containers shall be in accordance with the rules and regulations of the carrier applicable to the mode of transportation.

**5. 2. 4** Unless otherwise specified in the contract or order, Level C packing shall be provided.

**5. 3 Marking** - In addition to any special marking required by the contract or order, unit packages, intermediate packages, shipping containers for shipment to the Government, shall be marked in accordance with the requirements of MIL-STD-129.

## **6. NOTES**

**6. 1 Intended use** - This material is intended primarily for use in non-oxidizing fuel tank applications on space vehicles.

**6. 2 Ordering data** - Orders or contracts should specify the following information:

(a) Title, number, and change letter of this drawing.

(b) Dimensions of sheets or blocks.

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(c) Level of packaging and packing required.

(d) Options offered, if required (3.9, 4.1, and 5.2.4).

6.3 **CAUTION** - This material is not to be used in storage of oxidizing fluids. It shall be used only for storage of neutral or reducing cryogenic fluids.

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A | 18355 |

SCALE

REV

SHEET

## ADHESIVE, EPOXY BASE

### 1. SCOPE

1.1 Scope—This drawing covers the minimum requirements for one type of epoxy based adhesive applied by troweling and suitable for bonding metal to metal.

### 2. APPLICABLE DOCUMENTS

2.1 The following specifications and standards (and subsidiaries thereof), drawings and publications of issue in effect on date of invitation for bid, except as otherwise noted or controlled on an individual basis, form a part of this drawing to the extent specified herein.

### SPECIFICATIONS

#### FEDERAL

PPP-B-585	Boxes, Wood, Wirebound
PPP-B-601	Boxes, Wood, Cleated-Plywood
PPP-B-621	Boxes, Wood, Nailed and Lock-Corner
PPP-C-96	Cans, Metal, 28 Gage and Lighter
PPP-C-843	Cushioning Material, Cellulosic

#### MILITARY

MIL-A-5090	Adhesives, Heat Resistant, Airframe Structural, Metal to Metal
MIL-H-6088	Heat Treatment of Aluminum Alloys, Process For
MIL-S-8802	Sealing Compound, Temperature Resistant, Integral Fuel Tanks and Fuel Cell Cavities, High Adhesion
MIL-A-8920	Aluminum Alloy Plate and Sheet, ??19
MIL-B-10377	Box, Wood, Cleated, Veneer, Paper Overlaid
MIL-G-21380	Grit, Abrasive, Blasting

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## STANDARDS

### FEDERAL

Federal Test Method Standard No. 175

Adhesives, Methods of Testing

Federal Test Method Standard No. 406

Plastics, Method of Testing

### MILITARY

MIL-STD-129

Marking for Shipment and Storage

MIL-STD-401

Sandwich Constructions and Core Materials; General Test Methods

The following statement is applicable only for direct Government contracts:  
Copies of specifications, drawings and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting activity.

2.2 Other publications—The following publications form a part of this drawing.  
Unless otherwise indicated, the issue in effect on date of invitation for bid shall apply.

### 3. REQUIREMENTS

3.1 Preproduction approval—When preproduction approval is required by the contract or order, the material furnished under this drawing for preproduction approval shall be a product which has been tested and has passed the preproduction tests specified herein. After preproduction approval, the properties and method of manufacture shall not be changed without written approval from the procuring activity.

3.1.1 Preproduction sample—When preproduction approval is required, samples submitted shall be for preproduction tests as outlined in this drawing. Unless otherwise specified, one quart of base, as specified in the order or contract, with correct proportions of hardener to mix per manufacturer's recommendations, shall be supplied. All samples shall be identified with the title, number, and change letter of this drawing, base or hardener as applicable, manufacturer's name and designation, date of manufacture and mixing instructions.

3.2 Material—The materials used in the manufacture of the adhesive shall be of uniform high quality and readily mixed.

3.3 Application—The adhesive shall be applied by troweling.

3.4 Adhesive—The adhesive shall consist of two components designated as an epoxy base and hardener.

3.4.1 Base—The solids content of epoxy base shall be a minimum of 99 percent by weight.

3.4.2 Hardener—The solids content of hardener shall be a minimum of 99 percent by weight.

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**3.5 Cure**—The adhesive shall be capable of being cured at  $52 \pm 3^\circ\text{C}$  ( $125 \pm 5^\circ\text{F}$ ) for 16 hours under vacuum bag pressure of 50.8 cm (20 in.) minimum of mercury, followed by a post-cure at  $149 \pm 6^\circ\text{C}$  ( $300 \pm 10^\circ\text{F}$ ) for 32 hours without pressure.

**3.6 Mixing**—Complete mix of the two components shall be easily accomplished.

**3.7 Tensile bond strength**—The tensile bond strength to reinforced polyurethane foam per TBD shall not be less than the requirements of Table I and Table II.

TABLE I

Test Temperature $^\circ\text{C}$ $(^\circ\text{F})$	Tensile Bond Strength $\text{MN/m}^2$ (psi), minimum, Average of 5 specimens	Tensile Bond Strength, $\text{MN/m}^2$ (psi), minimum, of any one specimen
$-196 \pm 3$ ( $-320 \pm 5$ )	$1.1 \text{ MN/m}^2$ 150 psi	$0.69 \text{ MN/m}^2$ 100 psi

TABLE II

Tensile Load	Minimum Temperature at which Failure Occurs, average of 5 specimens	Minimum Temperature at which Failure Occurs, any one specimen
$0.7 \text{ MN/m}^2$ (100 psi)	$177^\circ\text{C}$ ( $350^\circ\text{F}$ )	$144^\circ\text{C}$ ( $300^\circ\text{F}$ )

**3.8 Specific gravity**—The cured adhesive shall have a specific gravity of 1.00 - 1.20.

**3.9 Storage stability**—Storage life of the unmixed components shall be a minimum of 6 months from date of manufacture when stored at  $25^\circ\text{C} \pm 6^\circ\text{C}$  ( $77^\circ\text{F} \pm 10^\circ\text{F}$ ). Unless otherwise specified in the contract or order, no material shall be supplied with a storage life of less than 5 months.

**3.10 Product marking**—The container of the production material shall be marked with the title, number and change letter of this drawing, manufacturer's name and designation, date of manufacture, batch number, base or hardener, mixing instructions and "STORE AT  $25^\circ\text{C} \pm 6^\circ\text{C}$  ( $77^\circ\text{F} \pm 10^\circ\text{F}$ )."

**3.11 Workmanship**—The material shall be uniform in quality and condition, free from inclusions, sediment, foreign material and other defects detrimental to fabrication or performance.

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MCDONNELL DOUGLAS

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#### 4. QUALITY ASSURANCE PROVISIONS

4.1 The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the procuring activity. Inspection records of the examination and tests shall be kept complete and available to the procuring activity as specified in the contract or order. The procuring activity reserves the right to perform any of the inspections set forth in the drawing where such inspections are deemed necessary to assure that supplies and services conform to prescribed requirements.

4.2 Lot—For purposes of sampling, inspection and tests, a lot shall consist of the material compounded at one time in one batch and offered for acceptance.

##### 4.3 Sampling -

4.3.1 Examination—Each lot of material shall be visually inspected to determine conformance to the applicable requirements of this drawing.

##### 4.4 Classification of tests -

4.4.1 Preproduction—Preproduction tests enable the procuring activity to determine that the material complies with the drawing requirements.

4.4.1.1 Tests—Samples shall be subjected to all of the tests described or referred to in 4.5 and 4.6 of this drawing.

4.4.2 Production—Production tests are those tests which shall be conducted by the manufacturer to assure conformance to the requirements of this drawing and enable the manufacturer to certify the accuracy of his product to the procuring activity.

4.4.2.1 Tests—The following tests shall be performed on samples from each lot of material and test records shall be kept in accordance with 4.1.

##### Paragraph

(a) Tensile bond testing

4.5.2

##### 4.5 Test methods -

4.5.1 Base—The solids content of epoxy base shall be a minimum of 99 percent when determined by testing in accordance with the requirements of MIL-S-8802.

4.5.1.1 Hardener—The solids content of the hardener shall be a minimum of 99 percent when determined by testing in accordance with the requirements of MIL-S-8802.

##### 4.5.2 Tensile bond testing -

4.5.2.1 Cryogenic test procedure—Five specimens prepared per 4.5.3 shall be tested at  $-196 \pm 3^\circ\text{C}$  ( $-320 \pm 5^\circ\text{F}$ ) per MIL-STD 401 to determine conformance to Table I.

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4.5.2.2 Elevated temperature test procedure—Five specimens shall be loaded at 0.7 MN/m<sup>2</sup> (100 psi) in a test fixture as shown in Figure A-1. One face of the specimen shall be heated to 177C (350F) and the temperature at which failure occurs shall be recorded to determine conformance to Table II.

#### 4.5.3 Specimen preparation -

4.5.3.1 Test panels—Aluminum alloy sheet shall be per MIL-A-8920 class T87 and anodized. The panels shall be 0.17 x 5.1 x 5.1 cm (0.069 x 2 x 2 in). The reinforced foam per TBD shall be 2.5 x 5.1 x 5.1 cm (1 x 2 x 2 in) with the "Z" direction parallel to the thickness.

4.5.3.2 Bonding of test panels—Adhesive shall be applied to the foam surface such that each yarn end has a fillet of adhesive surrounding it. The cleaned aluminum panel shall be placed on the faying surface of the foam.

4.5.3.3 Cure of adhesive—The test specimens shall be held for 17 hours at 25 ± 3C (77 ± 5F) at atmospheric pressure. The specimens shall then be cured at 52 ± 3C (125 ± 5F) for 16 hours + 15 minutes under vacuum bag pressure of 50.8 cm (20 in) minimum of mercury. The specimens shall be postcured at 149 ± 6C (300 ± 10F) for 32 hours minimum at atmospheric pressure.

4.5.4 Specific gravity testing—The specific gravity shall be determined per Federal Test Method Standard No. 406 at 25C ± 3C (77F ± 5F) and shall be recorded for each of 5 specimens.

4.5.4.1 Preparation of specimens—The test specimens shall be prepared by potting a 0.3 x 2.5 x 2.5 cm (1/8 x 1 x 1 in.) specimen against a suitable release material and curing per 4.5.4.3.

#### 4.6 Examination

4.6.1 Inspection for storage stability—The material shall be tested to establish conformance with the requirements of 3.10.

4.6.2 Inspection of packaging—The supplier shall make such inspections as are necessary to assure that the requirements for preservation, packaging, packing and marking are met.

#### 5. PREPARATION FOR DELIVERY

5.1 Packaging—Unless otherwise specified in the contract or order, the base shall be packaged in the following containers: 1/2 pint, 1 pint, 1 quart and 1 gallon. Hardener shall be packaged separately in the correct proportion to the base.

5.1.1 Unit packaging—Base may be furnished in tinplate cans, or glass jars with metal caps containing aluminum foil coated innerseals. If seams of cans or pails are soldered, they shall be coated with a suitable coating which is non-reactive to the base. Hardeners and accelerator may be furnished in glass ampules or Type V, Class 2 tinplate containers which will meet the tinplate and interior coating requirements of PPP C-96. Terneplate containers shall not be used.

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**TEST PROCEDURE:**

1. ASSEMBLE SPECIMEN IN FIXTURE
2. POSITION HEATER ELEMENT PLATE FLAT AGAINST SPECIMEN BLOCK
3. LOAD IN TENSION TO 1,780 N (400 LB) 0.69 MN/m<sup>2</sup> (100 PSI)
4. ADJUST POWER TO HEATER TO REACH ~20°C (~400°F) ON THERMOCOUPLE INSIDE SPECIMEN BLOCK WITHIN 4 TO 5 MINUTES
5. RECORD TEMPERATURE AT RUPTURE

**NOTE:** DISCARD SPECIMENS THAT RUPTURE PRIOR TO APPLICATION OF HEAT OR THAT RUPTURE PRIOR TO TEMPERATURE INDICATION OF 93°C (200°F)

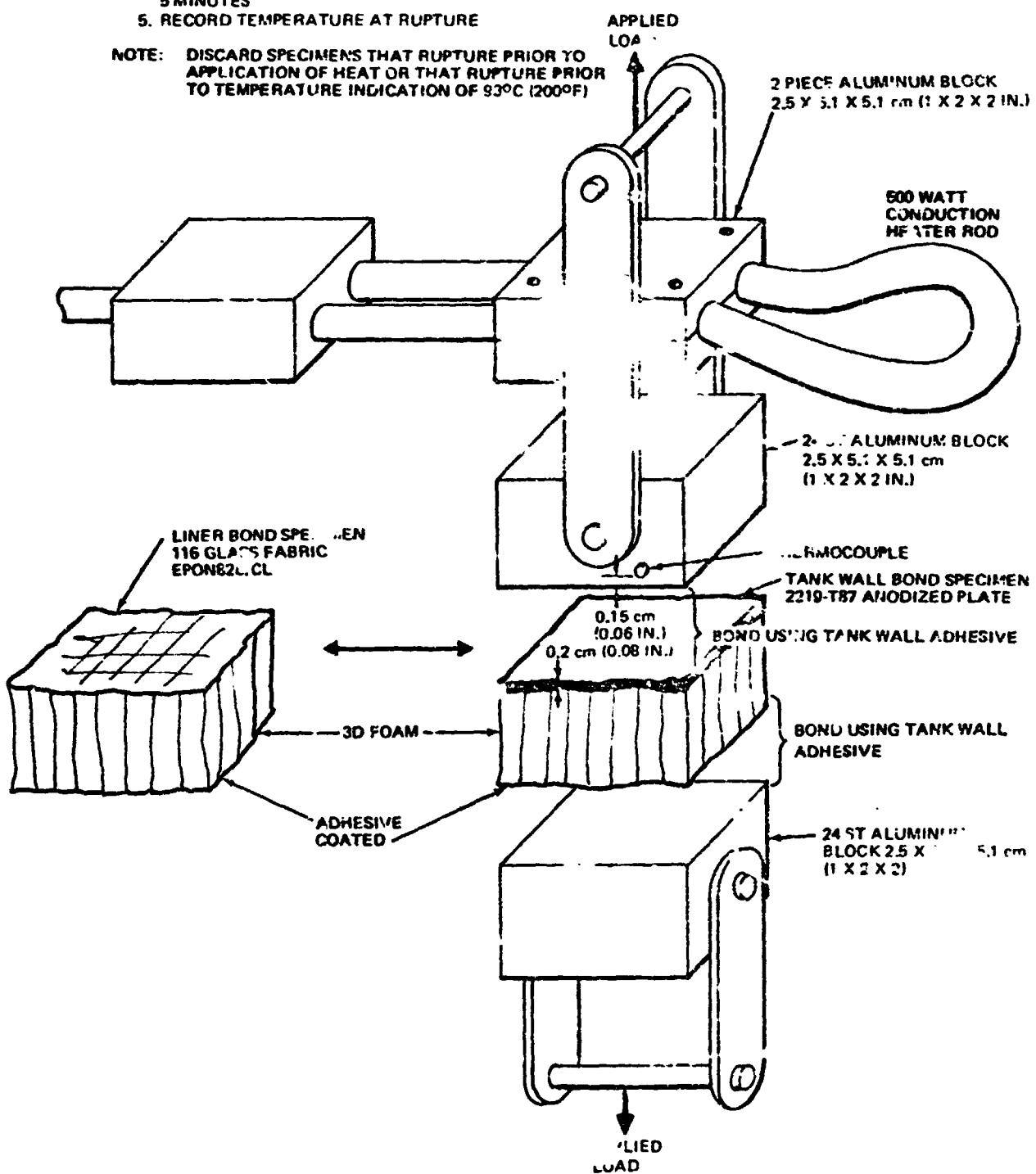


Figure A-1. Elevated Temperature Test Fixture Tensile Bond Strength – Hot Plate

5.1.2 Level C-Packaging shall be in accordance with the manufacturer's commercial practice.

5.1.3 Unless otherwise specified in the contract or order, Level C preservation and packaging shall be provided.

5.2 Packing -

5.2.1 Cushioning-Unless otherwise specified by the procuring activity, when unit fiberboard boxes are required, the contents shall be secured snugly by means of vertical separators and fiberboard or corrugated paper to fill voids. Cushioning material shall conform to PPP-C-842. The fiberboard shall be made of single or double wall corrugated board and the corrugated paper shall be flexible and single faced.

5.2.2 Level A-Unit packages shall be packed in wood cleated plywood (overseas type), nailed wood or wirebound wood (Style 3 for Type 2 load) boxes conforming to PPP-B-601, PPP-B-621 or PPP-B-585, respectively. Box closures shall be as specified in the applicable box specification appendix. The weight of wood boxes and contents shall not exceed 200 pounds.

5.2.3 Level B-Unit packages shall be packed in wood cleated fiberboard, nailed wood or wirebound wood (for Type 1 load) corrugated or solid fiberboard, wood cleated plywood (domestic type), or wood cleated paper or erlaid (domestic type) boxes conforming to PPP-B-621, PPP-B-585, or MIL-B-1637T, respectively. Closures shall be as specified in the box specification or appendix thereto. Fiberboard boxes shall conform to the special requirements of the applicable box specification.

5.2.4 Level C-When specified by the procuring activity, the unit packages shall be packed in containers in a manner to insure safe delivery and acceptance at destination. Containers shall comply with the rules and regulations of the carrier applicable to the mode of transportation.

5.2.5 Unless otherwise specified in the contract or order, Level C packing shall be provided.

5.3 Marking -

5.3.1 Marking of packages-Each unit package, can, ampule, and so forth, as applicable shall be marked in accordance with MIL-STD-129 and with the following notes:

**CAUTION:** Special care should be taken to avoid inhaling the activator fumes. Avoid contact of the epoxy resin or the accelerator with the skin.

**NOTE:** Uncured epoxy resin or accelerator may be removed from contact area by washing with toluol or acetone, then with soap and water.

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MILITARY AIRCRAFT DIVISION  
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5.3.2 Marking of shipping containers - In addition to any special marking required by the contract or order, shipping containers shall be marked in accordance with MIL-STD-129 and with the following note on storage.

**STORAGE:** The material called for in this drawing should be stored at temperatures from 19°C to 31°C (67°F to 87°F) in the absence of sunlight. If exposed to temperatures in excess of 31°C (87°F) for prolonged periods, the storage life of the material will be proportionately reduced.

6. NOTES

6.1 Intended use - The epoxy adhesive covered by this drawing is intended for use in the adhesion of cryogenic insulation where external temperature may reach 177°C (350°F).

6.2 Ordering data - Orders or contracts should specify the following information:

- (a) Number, title, base or hardener, date of manufacture, manufacturer's name and designation and change letter of this drawing
- (b) Size of container and level of packaging and packing (see 5.1)
- (c) Options offered, if required (see 3.1, 4.1, 5.1.3 and 5.2.5)

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HUNTINGTON BEACH, CALIF.

MCDONNELL DOUGLAS

SIZE CODE IDENT NO. DRAWING NO.

A 18355

SCALE

REV

SHEET

INSULATION, INTERNAL, INSTALLATION OF  
(FOR NON-OXIDIZING SYSTEMS)

1. SCOPE

1.1 Scope - This drawing covers the minimum requirements for the installation of insulation to the interior of tanks of non-oxidizing systems which will be exposed to temperatures in the range of -253C (-423F) to 177C (350F).

2. APPLICABLE DOCUMENTS

2.1 Government-furnished documents - The following documents (and subsidiaries thereof), in issue in effect on date of invitation for bid, unless otherwise indicated, form a part of this drawing to the extent specified herein.

SPECIFICATIONS

MILITARY

MIL-C-9084

Cloth, Glass, Finished, For Polyester Resin Laminates

MIL-R-9300

Resin, Epoxy, Low Pressure Laminating

STANDARDS

MILITARY

MIL-STD-401

Sandwich Constructions and Core Materials, General Test Methods

Copies of specifications, standards, drawings, and publications required by contractors in connection with specific procurement functions should be obtained from the procuring activity or as directed by the contracting activity.

2.2 Other publications - The following publications (and subsidiaries thereof) form a part of this drawing to the extent specified herein. Unless otherwise specified, the issue in effect on date of invitation for bid shall apply.

AMERICAN SOCIETY FOR TESTING AND MATERIALS

ASTM D 676

Methods of Test for Indentation of Rubber by Means of a Durrometer

(Application for copies of ASTM standards should be addressed to the American Society for Testing and Materials, 216 Race Street, Philadelphia, Pennsylvania 19103).

(LFR KOWELD 211 A/I.Z.)  
(New Specification)

Adhesive, Epoxy Base, High Temperature

McDONNELL DOUGLAS ASTRONAUTICS CO.  
McDONNELL DOUGLAS CORP.  
McDONNELL DOUGLAS CORP.

500-212-1202 (06 AUG 71)

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### 3. REQUIREMENTS

3.1 Materials - The tank materials used for bonding, core insulation, and interior lining shall be as specified on the design drawing.

3.1.1 Core-to-wall adhesive - Adhesive for bonding insulation core to the tank wall shall be Lefkoweld 211 A/Z or equivalent.

3.1.2 Liner-to-core adhesive - Adhesive for imprinting glass cloth liner and bonding to the insulation core shall be per MIL-R-9300 Type I, Class 1, or equivalent.

3.1.3 Adhesive validation - Prior to use, the adhesives shall be stored under controlled conditions to ensure continued compliance with the respective specifications.

3.1.4 Liner - The glass cloth liner shall be per MIL-C-9084, Type II.

3.2 Cleaning - The tank interior shall be cleaned free from particles and foreign materials and shall be suitable for adhesive bonding. The cleaning operation shall not degrade the integrity of the structure.

3.3 Bonding environment - The bonding environment shall have controlled temperature and humidity as necessary to prevent the collection of visible moisture on the bonding surfaces and as necessary to prevent degradation of the adhesive bond. The bonding shall be conducted in an area that has filtered air and is free of contaminants to the extent that bonding operations are not affected.

### 3.4 Bonding Procedure -

#### 3.4.1 Process control -

3.4.1.1 Cryogenic testing - The process shall be controlled by bonding panels for strength specimens simultaneously with production parts. Tensile bond strength of the insulation core-to-tank wall and liner-to-insulation core shall be a minimum of 0.69 MN/m<sup>2</sup> (100 psi) at -196°C (-320°F); and a minimum average of 1.1 MN/m<sup>2</sup> (150 psi) at -196°C (-320°F).

3.4.1.2 High temperature testing, core-to-tank bond - The strength specimens shall be bonded simultaneously with production parts. The temperature at which tensile bond failure under a stress of 0.69 MN/m<sup>2</sup> (100 psi) occurs shall be on the average of 5 samples in excess of 177°C (350°F), with a minimum of 149°C (300°F) for only one sample. Use elevated temperature test fixture, tensile bond strength, as shown in Figure A-1.

3.4.1.3 High temperature testing, liner-to-insulation core bond - The strength specimens shall be bonded simultaneously with production parts. The temperature at which tensile bond failure under a stress of 0.69 MN/m<sup>2</sup> (100 psi) occurs shall be on the average of 5 samples in excess of 163°C (325°F), with a minimum of 141°C (285°F) for any one sample. Use elevated temperature test fixture tensile bond strength as shown in Figure A-1.

3.4.2 Adhesive mixing - The adhesive and hardener components shall be mixed in accordance with the applicable material specification. The mixed adhesive shall be applied, the parts assembled, and the required bonding pressure applied before the adhesive work life is expired, as shown in Figure A-2.

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**TEST PROCEDURE:**

1. ASSEMBLE SPECIMEN IN FIXTURE
2. POSITION HEATER ELEMENT PLATE FLAT AGAINST SPECIMEN BLOCK
3. LOAD IN TENSION TO 1,730 N (400 LB) 0.69 MN/m<sup>2</sup> (100 PSI)
4. ADJUST POWER TO HEATER TO REACH ~204°C (~400°F) ON THERMOCOUPLE INSIDE SPECIMEN BLOCK WITHIN 4 TO 8 MINUTES
5. RECORD TEMPERATURE AT RUPTURE

**NOTE:** DISCARD SPECIMENS THAT RUPTURE PRIOR TO APPLICATION OF HEAT OR THAT RUPTURE PRIOR TO TEMPERATURE INDICATION OF 23°C (200°F)

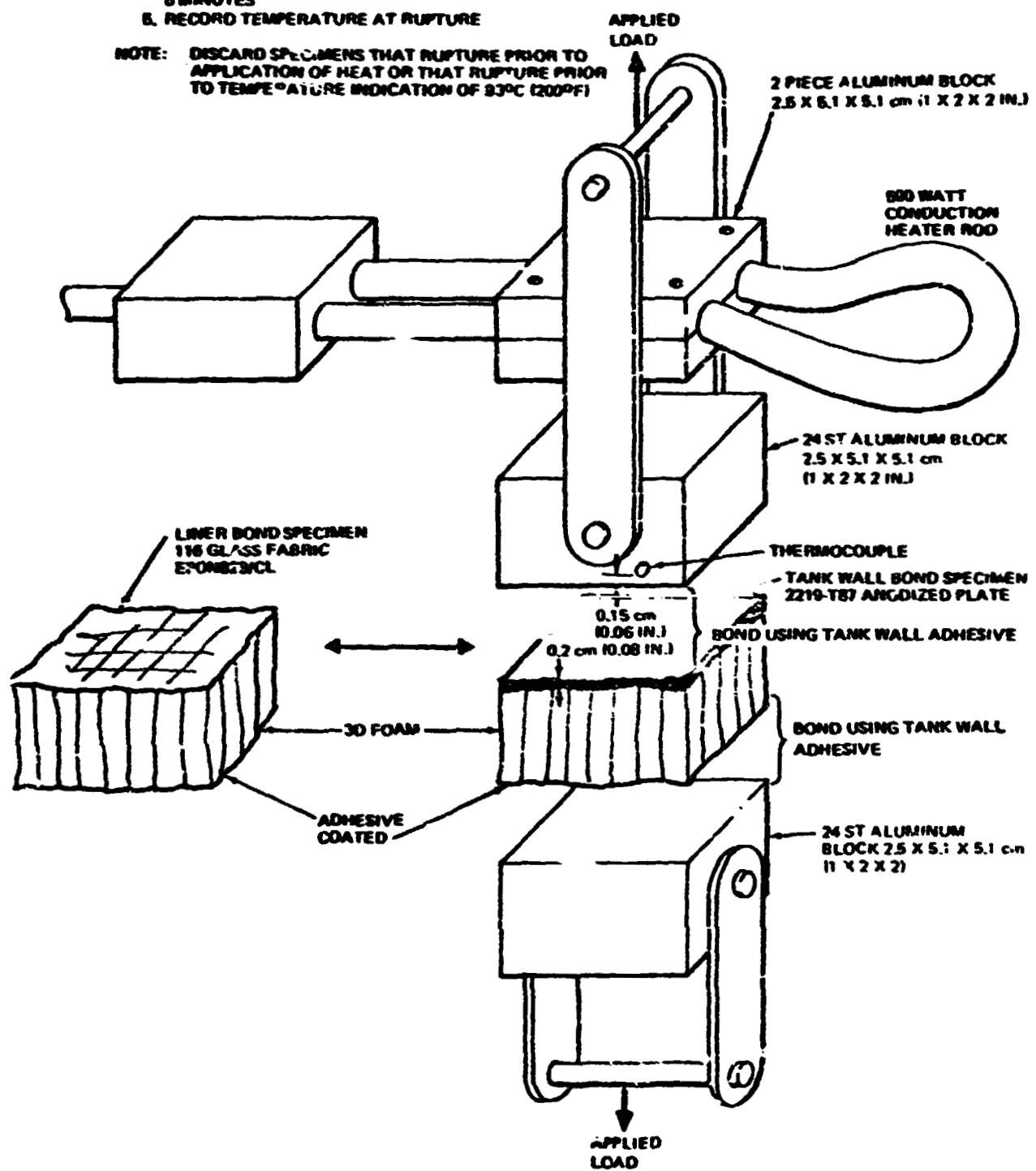


Figure A-1. Elevated Temperature Test Fixture Tensile Bond Strength – Hot Plate

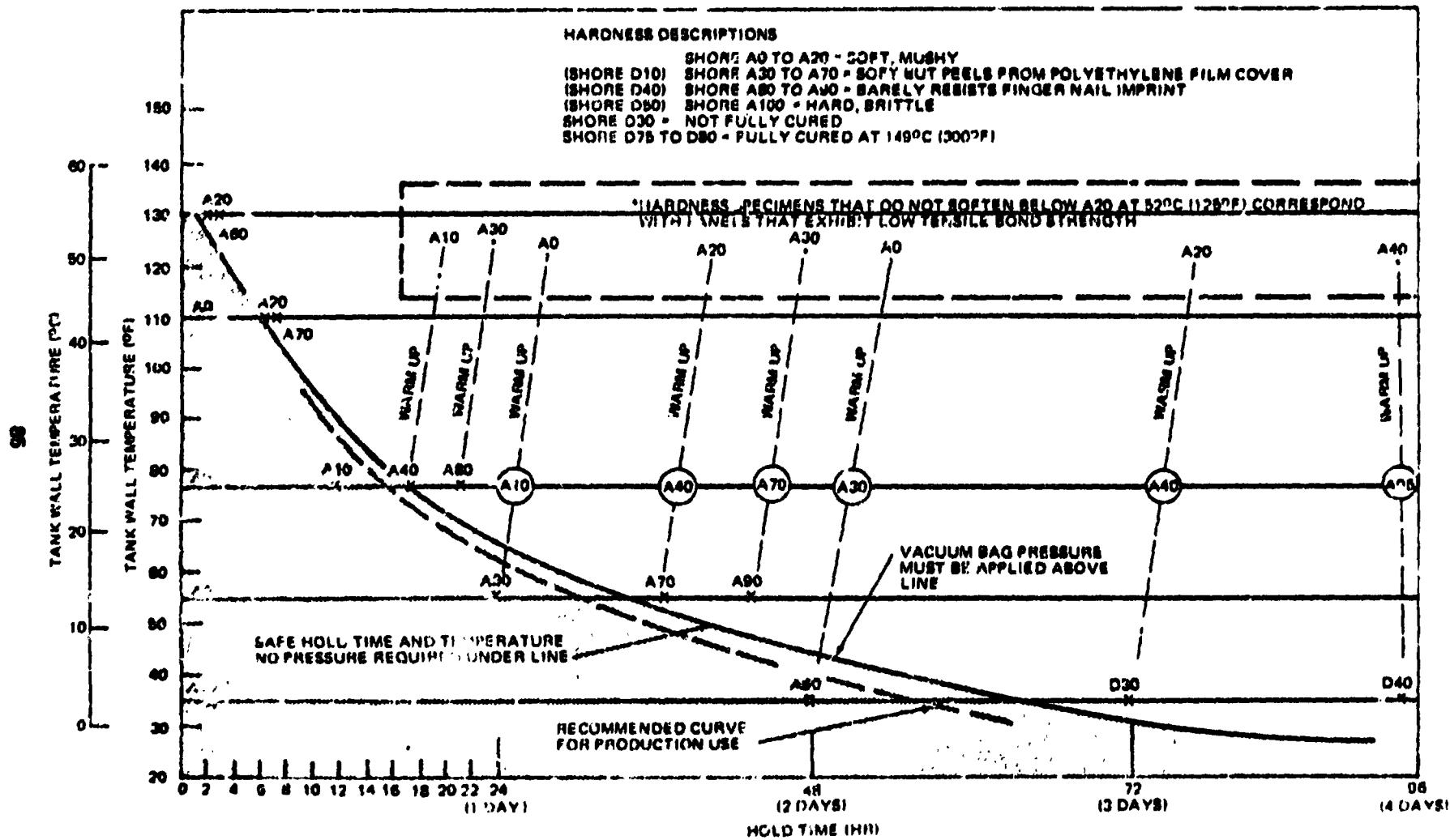


Figure A-2. Catalyzed Working Life of Tank Wall Adhesive

**3.4.3 Adhesive application** – The adhesive shall be applied such that each yarn end has a fillet of adhesive surrounding it.

**3.4.3.1 Adhesive weight - (core to tank) -** After application, the adhesive weight shall be 0.28 to 0.30 kg/m<sup>2</sup> (26 to 28 g/ft<sup>2</sup>) of surface area for insulation core-to-tank wall bond.

**3.4.5.2 Resin weight - MIL-R-9300 Type I, Class I** – The weight of resin used to impregnate and bond the glass cloth liner to the insulation core shall be  $0.16 \pm 0.1^2 \text{ kg/m}^2$  ( $15 \text{ g/ft}^2$ ) of liner area. Local areas, such as around fittings, inserts, vent, or access holes, are exempt from liner adhesive weight limitations. However, the liner in these localized areas must conform to workmanship requirements of this drawing. The catalyzed working life of the liner resin shall be followed as shown in Figure A-3.

**3.4.3.3 Weight of liner porosity seal coat** - The weight of MIL-R-9300 Type I, Class I adhesive remaining on the liner surface after sealing operations shall be 33 gm to 55 gm/m<sup>2</sup> as determined by visual comparison with previously prepared panels of identical configuration to the production liner. Resin runs or other local resin accumulations shall be included in the total weight restriction.

3. 4 Gap filling — Gaps shall be filled as required by the design drawing. Gap filler shall consist of the following:

- 0.100 parts by weight of BJO-0930 phenolic microballoons  
0.25 parts by weight of 0.064 cm (0.25 in.) milled glass fibers  
0.300 parts by weight of MIL-R-9300 Type I, Class L

### **3. 4. 5 · Standard care cycles –**

**3.4.5.1 Cure - (core to tank and liner to cor.)** - The adhesive shall be cured by the following standard cure, or an equivalent cure as necessary to meet the requirements of the applicable design:

**Cure at temperature of 52C ± 1°C (125F ± 10F) under vacuum bag pressure of 20 inches minimum of mercury for 16 hours.**

**Follow by post curing using the following stepwise cure temperatures.**

1 hour minimum at 66C  $\pm$  5C (150F  $\pm$  10F)  
1 hour minimum at 82C  $\pm$  5C (180F  $\pm$  10F)  
1 hour minimum at 93C  $\pm$  5C (200F  $\pm$  10F)  
1 hour minimum at 121C  $\pm$  5C (250F  $\pm$  10F)  
32 hours minimum at 149C  $\pm$  5C (300F  $\pm$  10F)

**3.5 Workmanship** — Before mating, the adhesive shall be free of adulterants, voids, and air bubbles detrimental to the adhesive bond. Foreign materials, such as tape or bag sealing compound remaining in the composite insulation shall be irrigated with articles lodged firmly in crevices of the cable joints or in crevices (such as irregularities; foreign materials shall individually cover the area

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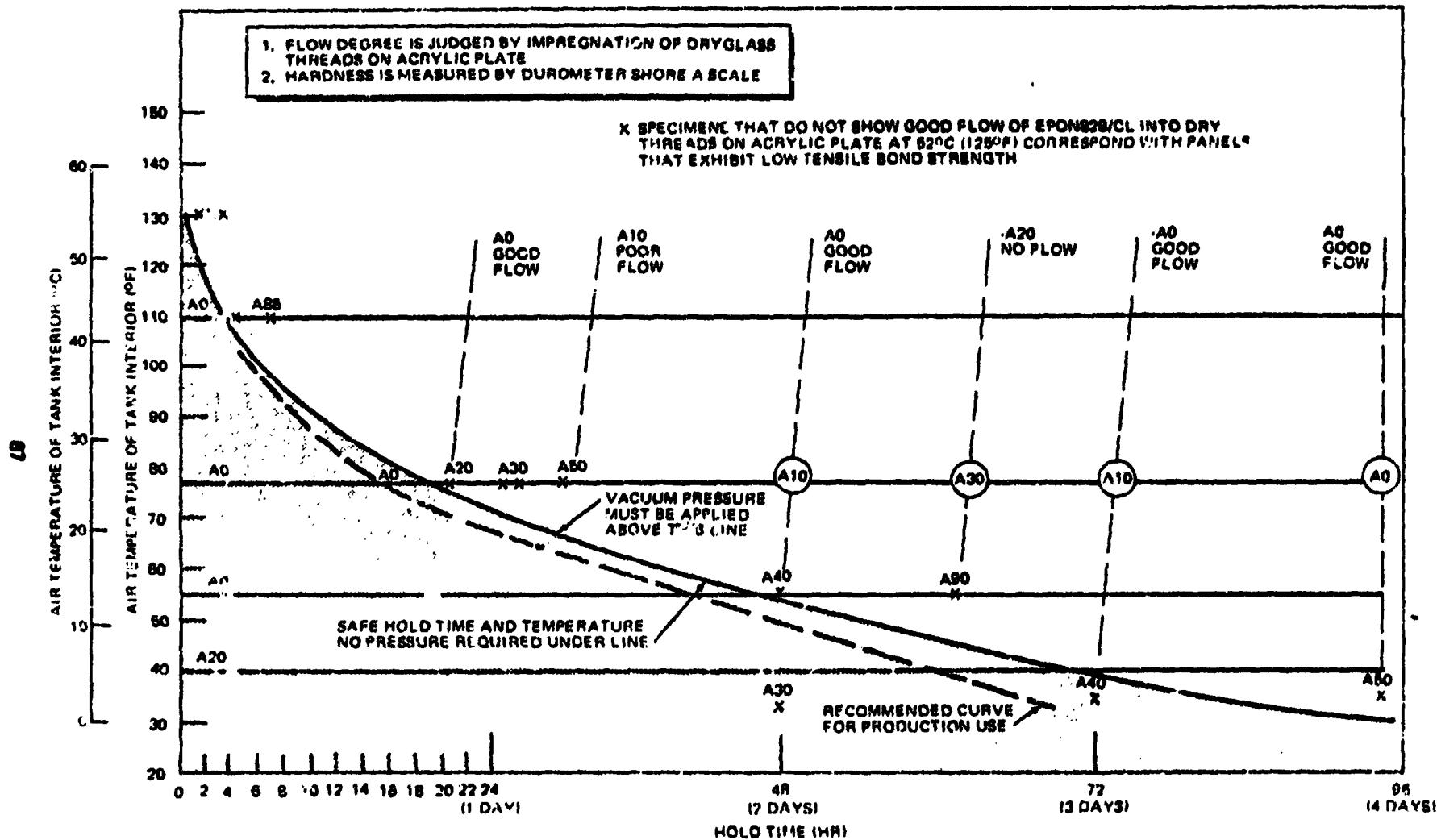


Figure A-3. Catalyst Working Life of Liner Resin

no greater than  $0.81 \text{ cm}^2$  ( $0.125 \text{ in.}^2$ ). Localized particles of lint, foam dust, or cloth strings shall be firmly embedded in the adhesive and shall not protrude above the surface of the liner more than  $0.32 \text{ cm}$  ( $0.125 \text{ in.}$ ). The quality of the glass liner and the bond between liner and insulation core shall meet the following requirements:

- (a) Cloth wrinkles or excess resin ridges shall be limited to  $0.32 \text{ cm}$  in height.
- (b) There shall be no unbond areas in liner overlap areas larger than  $6.45 \text{ cm}^2$  ( $1 \text{ in.}^2$ ) per  $30.5 \text{ cm}$  ( $1 \text{ ft.}$ ) of overlap length and shall not extend through more than one-half the width of the overlap. These unbonded areas shall be contained within the overlap width, and shall not run out at the edge leaving loose edges of liner. Liner overlap requirements shall include the joints at fittings and liner-to-tank wall termination joints.
- (c) When glass-thread-reinforced insulation core is specified in the design drawing, the unbonded areas between the liner-to-insulation core shall be identified as being separated from the vertical glass thread reinforcement in the insulation core. Single areas of unbond shall be limited to a group of nine threads maximum, or an area of  $1.27 \text{ cm}$  ( $0.5 \text{ in.}$ ) in diameter, whichever is the larger. The total of unbonded threads in any  $930 \text{ cm}^2$  ( $1 \text{ ft.}^2$ ) shall not exceed forty (40) or one percent of total threads per  $930 \text{ cm}^2$ . When other types of insulation core are specified on the design drawing, the unbond between liner and core shall not exceed  $1.27 \text{ cm}$  diameter and shall be limited to five such areas per  $930 \text{ cm}^2$ .
- (d) Unbonded areas of the liner under cloth wrinkles, or at offset insulation core joints shall not exceed  $0.64 \text{ cm}$  ( $0.25 \text{ in.}$ ) in width and  $30.5 \text{ cm}$  in length without intermittent contact with the insulation core.

#### 4. QUALITY ASSURANCE PROVISIONS

4.1 Inspection responsibility — The supplier is responsible for the performance of all inspection requirements as specified herein. Except as otherwise specified, the supplier may utilize his own or any other inspection facilities and services acceptable to the procuring activity. Inspection records of the examination and tests shall be kept complete and available to the procuring activity as specified in the contract or order. The procuring activity reserves the right to perform any of the inspections set forth in the drawing where such inspections are deemed necessary to assure that material and process conform to the prescribed requirements.

4.2 Lot — A lot shall consist of a single tank completely insulated and presented for inspection.

4.3 Sampling — Each lot shall be inspected 100 percent.

4.4 Classification of tests —

4.4.1 Production — Production tests are those tests which shall be conducted by the manufacturer to assure conformity to the requirements of this drawing and enable the manufacturer to certify the accuracy of the product to the procuring activity.

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Paragraph

- |   |         |
|---|---------|
| (a) Adhesive weight - (core to tank wall) | 4. 5. 1 |
| (b) Resin weight - (core to liner)        | 4. 5. 2 |
| (c) Hardness                              | 4. 5. 3 |
| (d) Process control                       | 4. 5. 4 |
| (e) Examination                           | 4. 6    |

**4. 5 Test methods and procedures -**

**4. 5. 1 Adhesive weight** - Three specimens shall be prepared by covering paper samples, at least 6 inches by 6 inches in size, with adhesive identical to that applied to the insulation core at the start of bonding each tank segment (see 6. 3. 1) and one specimen after every twentieth individual insulation core in the segment. These specimens shall be weighed and the weight per square foot calculated to verify the requirements of 3. 4. 4. 1 before the insulation core is installed.

**4. 5. 2 Resin weight** - MIL-R-9300 Type I, Class I - One specimen, a minimum of one square foot in area, shall be cut from each fifteen feet of continuous, adhesive-impregnated glass cloth; for lengths less than fifteen feet of adhesive-impregnated glass cloth a one-square foot specimen shall be cut from the end. These specimens shall be weighed and the weight of adhesive per square foot calculated to verify the requirements of 3. 4. 4. 2 before the sheets are installed.

**4. 5. 3 Process control** - To control the process of 3. 4. 1, representative control panels for tensile strength specimens shall be prepared for each bonding operation encompassed by one vacuum pressure bag or for each segment. One control panel shall represent the adhesive bonding operation insulation core-to-tank wall and one control panel shall represent the glass cloth liner-to-insulation bond.

**4. 5. 3. 1 Panel preparation** - Each panel shall consist of the same construction and materials as used in the corresponding tank segment. The metal plates simulating the tank wall shall be cleaned, primed, or otherwise prepared in the same manner and with the same batch of materials as used in the segment. The adhesive-coated components and the impregnated glass cloth liner shall be assembled to form the panel for process control specimens at the beginning of the segment bonding sequence and shall be cured under pressure at the same time and under the same environmental conditions as the corresponding segment. Post cure of the panels (no pressure) shall represent the post cure of the tank insulation. The post cure temperature shall be as specified in 3. 4. 6.

**4. 5. 3. 2 Specimen preparation** - Five tensile strength specimens shall be prepared from each control panel per 4. 5. 4 and 4. 5. 4. 1.

**4. 5. 3. 3 Tensile strength test** - Specimens prepared per 4. 5. 4. 2 shall be tested in accordance with MIL-STD-401 to determine conformance to the requirements of 3. 4. 1.

**4. 5. 3. 4 High temperature test** - Specimens prepared per 4. 5. 4. 2 shall be tested in accordance with MIL-STD-401, except as specified herein. Apply a tensile load of 0.49 MP<sup>2</sup> (100 psi) to the specimen. Apply heat to one face of the specimen to achieve a bond line temperature of 204C (400F) within 4 to 5 minutes. Measure the temperature at which failure occurs to determine conformance to the requirements of 3. 4. 1.

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4.6 Examination -- Each segment shall be visually inspected to determine conformance to the requirements of 3.2, 3.4.3.3, 3.4.4, and 3.5.

## 5. PREPARATION FOR DELIVERY

There are no applicable requirements.

## 6. NOTES

6.1 Intended use -- The installation of a cryogenic insulation is intended for use on the interior of tanks designed to store non-oxidizing cryogenic fluids at temperatures as low as -235C (-423F).

6.2 Ordering data -- Order or contract should specify the following:

(a) Title, number and change letter of this drawing.

## 6.3 Definitions --

6.3.1 Segment -- A segment is the sum of all individual pieces of insulation core, or glass liner, bonded and cured in one operation and under one vacuum pressure bag.

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